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Report on the Treatability Study for Inerting Small Quantities of Radioactive Explosives and Explosive Components

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Report on the Treatability Study for Inerting Small Quantities of Radioactive Explosives and Explosive Components

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Abstract

As a result of Sandia's radiation hardening testing on a variety of its explosive components, radioactive waste streams were generated and have to be disposed of as radioactive waste. Due to the combined hazards of explosives and radioactivity, Sandia's Radioactive and Mixed Waste Management organization did not have a mechanism for disposal of these waste streams. This report documents the study done to provide a method for the removal of the explosive hazard from those waste streams. The report includes the design of the equipment used, procedures followed, results from waste stream analog tests and the results from the actual explosive inerting tests on radioactive samples. As a result of the inerting treatment, the waste streams were rendered non-explosive and, thus, manageable through normal radioactive waste disposal channels.

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Acknowledgments

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Contents

Introduction.....	6
Definition of the Problem.....	6
Approach.....	7
Description of the Inerting Chamber and Ancillary Equipment	8
Chamber Design Qualification.....	9
Inerting Procedure.....	10
Discussion of Results.....	11
Temperature of the Chamber During Testing	11
Analog Tests	12
Radioactive Tests	13
Post-Test Survey of the Equipment.....	15
Conclusions and Recommendations	15
References.....	29
 APPENDIX A: Proof Test of Inerting Chamber.....	 1A
APPENDIX B: Operating Procedure Used in the Hot Cell Facility.....	1B

Figures

1 Schematic Figure of Inerting Chamber.....	19
2 Schematic Layout of Inerting Chamber and Ancillary Equipment	20
3 Temperature vs Time; Analog Tests 3, 4 and 5	21
4 Temperature vs Time; Analog Tests 6, 7, 8 and 9	22
5 Temperature vs Time; Analog Tests 10, 11 and 12	23
6 Temperature vs Time; Radioactive Tests 1, 2 and 3	24
7 Temperature vs Time; Radioactive Tests 4, 5 and 6	25
8 Thermally Decomposed MC4217 Detonators	26
9 Thermally Treated Polycarbonate Explosive Block	27
10 HNS Explosive Residue From Thermal Inerting Process.....	28

Tables

1 Description of Waste Streams.....	16
2 Thermal Characteristics and Inerting Requirements of Some Explosives	16
3 Description of Explosives Inerting Tests Performed on Non-Radioactive Analog Materials	17
4 Description of Explosives Inerting Tests for Radioactive Components.....	18

Report on the Treatability Study for Inerting Small Quantities of Radioactive Explosives and Explosive Components

Introduction

Sandia National Laboratories (SNL) has in the past subjected many of its weapon components, both explosive and non-explosive, to radiation hardness testing. In most cases those tests were carried out during the regularly scheduled underground weapons tests which were done at the DOE's Nevada Test Site; occasionally, however, some components were subjected to above ground testing in nuclear reactors, proton accelerators, or other suitable sources of the appropriate radiation. In any case, much of the testing resulted in components that became radioactive due to the activating nature of the radiation. In today's restrictive and regulated environment it has become difficult to properly dispose of such materials. As a result of this more restrictive environment, Sandia Laboratories' Waste Management Organizations have often found themselves in possession of waste streams for which they have no acceptable disposal methods or treatment technologies.

In the future Sandia's generation of such waste streams will be considerably reduced, but almost certainly not precluded. In addition, future Sandia efforts to properly treat and dispose of explosive mixed waste streams will almost certainly face regulations that are at least as restrictive as current ones. The treatability study documented below established a set of processes and conditions which efficiently converted an explosive, mixed waste stream to a non-explosive, mixed waste stream manageable by standard procedures. In doing so, the requirement(s) for storage, handling and transportation of the waste streams was considerably simplified and the total volume of the wastes reduced.

Definition of the Problem

The problem facing Sandia's radioactive waste management organization was disposal of the mixed waste streams which contained explosives. There are mechanisms for the safe disposal of both radioactive and treated mixed waste. There are, however, no mechanisms for the safe and compliant disposal of mixed wastes which contain explosives. In order for these waste streams to be properly disposed of they first had to be made to meet Land Disposal Restrictions (LDRs); i.e., rendered safe and non-explosive. Thus, a method for removing the explosive characteristic from those waste streams needed to be developed.

Several possible approaches for removal of the explosive characteristic, i.e. deactivation, were considered. These included solvent extraction, solvent extraction followed by chemical reduction of the explosive, solvent extraction followed by incineration in the solvent, small scale molten metal bath destruction and thermal destruction. The solvent extraction methods were rejected because they generated additional hazardous secondary waste. The molten metal bath, also a thermal treatment method, was rejected because it was difficult to implement.

The method chosen for inerting these explosives was based on the DOE's "Explosive Safety Manual"¹ recommendation for "decontaminating" items that are difficult to clean by normal washing or extraction techniques. For those items that contain "...cracks, seams, voids, or

other places where explosive residue may be inaccessible..." the safety manual recommends a thermal method. The safety manual requires that "Final decontamination by thermal techniques shall be done by subjecting the item to sustained heating at a temperature at least 60 °C higher than required for decomposition of the most thermally stable explosive substance present. The item shall be kept at that temperature for a sufficient period of time to ensure that all parts have reached that temperature and all explosives material is decomposed." ^{1a} In dealing with radioactive or mixed waste the term "decontamination" typically refers to the removal of the radioactive characteristic from the waste. The DOE manual, however, refers to the removal of the explosive characteristic as "decontamination". In order to avoid confusion, in this report we will refer to the removal of the explosive characteristic as "inerting".

A thermal method was chosen for two reasons: first, this method is most suitable for treating hardware, equipment and other solid structures that are difficult to handle and inspect; second, this method is appropriate when waste stream minimization is an important goal. Waste minimization is always an important goal in Sandia's operations today; in operations involving radioactive materials it becomes even more important.

The waste streams used in this treatability study are described in Table 1. With the exception of stream 930192-1, the radioactivity in these waste streams was due to neutron activation of metals. Consequently, the activity was bound in either a metal or metal oxide matrix and not volatile. In the case of 930192-1, there was uncertainty regarding whether there was any contamination at all. This waste stream was generated during a clean-up operation following an incident in which an HNS pellet was exposed to an atmosphere containing a trace of ³H. No confirmation of contamination was obtained because liquid scintillation analysis of the explosive would be required and would result in an additional mixed waste sample being generated. This sample was assumed to be radioactive and treated as such.

The largest quantity of explosives was in stream number 910402-1 which contained approximately 32 grams of Pentaerythritoltetranitrate (PETN). In this case, however, the explosives were present in subsets of approximately 2.5 gm; and could be subdivided for treatment. The material with the highest requirement for bakeout was a blend of Titanium and Potassium Perchlorate (TiKP) in waste stream 940025. This material is not a detonable explosive, it is a gas producing pyrotechnic. It requires a bakeout temperature of 490 °C, see Table 2, in order to meet the requirements of the DOE's Explosive Safety Manual.

Approach

Two approaches to a thermal inerting process were considered. The first option was to purchase an explosion proof, high temperature oven or autoclave and to make any modifications needed to permit the treatment of the radioactive materials. This option was a relatively high cost option for the following reasons. First, a facilities modification work order would probably be required to have an oven installed in Area II, where the feasibility tests would be conducted. Second, when feasibility tests in Area II were complete and processing procedures developed, the oven would have to be moved to the Hot Cell Facility (HCF) in

Area V for the radioactive work. That move might also require some facilities modifications in the HCF. Third, explosion proof ovens are relatively high cost items.

The second lower cost option considered was to design and build a small, heavy-walled steel cylinder, which will hereafter be referred to as the inerting chamber, with the capability to safely contain the explosion of a small quantity of explosives. The DOE Explosives Safety Manual is quite specific regarding requirements for equipment used to heat explosives; these requirements include such fail safe features as temperature override shutoff, dual controls and constant monitoring during operation.

These requirements are exempted, however, for "Systems capable of total containment of the effects of an explosion..."^{1b}. This exemption provided a mechanism for designing a relatively low cost inerting chamber for this study. This chamber would need to be designed with the capability for heating small quantities of explosives inside of it and still be small enough to fit inside a conventional fume hood. The fume hood would be used to provide an acceptable vent for gaseous decomposition products of the explosives; as well as to provide a suitable exhaust in the event that an inerting test were to leak some radioactivity. Fume hoods with HEPA and other filtering for containing small radioactive releases are available in Area V.

Given that the waste streams of concern were all either small quantities, or separable into small quantities, of explosives and small components and that future waste streams would most probably also be small, the small chamber option was selected as the one of choice. The design of the chamber is described in drawings R45632, R45633, R45634, R45652, R45659 and R45660 and is discussed in more detail later in this report. The drawings are in the SNL drawing control system and under formal change order requirements. A brief description of the chamber and ancillary equipment is given below.

Description of the Inerting Chamber and Ancillary Equipment

The chamber (Figure 1), including closures and bolts, is machined from 17-4PH stainless steel and hardened to condition H1025. At that condition this material retains most of its strength to temperatures as high as ~ 370 °C and is thus well suited for this application. The chamber was formed by the cylinder, which is 18" long with an 8" I.D., and two heavy walled, machined end covers. Each cover was attached to the cylinder with eight 1/2" bolts. One end cover was blank and was removed every time a sample needed to be installed or removed. The second cover was machined with male pipe thread penetrations to provide a mechanism for (1) evacuating and purging the chamber as necessary, (2) electrical feed throughs for heating elements and (3) feed throughs for thermocouples. This end plate was not removed once it was installed. The end plates were sealed to the cylinder using conflat style sealing rings made of copper.

For the inerting operations the materials to be processed were placed in a covered, rectangular stainless steel tray connected to three heating elements (350 watt, stainless steel covered heating strips, Watlow P/N S2JBVI). The elements were attached to the sides and bottom of the tray. The processing tray was held approximately centered in the cylinder by a set of legs that had been formed by attaching to the tray four machine screws. The tray stood on a flat, 3/16" thick steel plate (~3"xl2") that was inserted into the cylinder to provide a flat surface for

the tray. The tray needed to be extractable from the cylinder without breaking electrical disconnections to the heaters in order to facilitate the loading and unloading of the materials to be processed. This feature required that the wires from the feed throughs to the heating elements be considerably longer than the length of the cylinder; the excess wire was accommodated by folding it into place under the flat steel plate. Pieces of woven-glass fire blanket were used as insulation between the processing tray and the cylinder walls; this insulation was installed after the tray was loaded and installed. This insulation served to reduce radiant heating of the cylinder during the inerting process. Data to be presented later show that a significant difference in sample and wall temperatures was observed during these operations.

The temperature of the samples was controlled by an Omega MCS-5001 temperature controller modified by the installation of a 25 amp solid state relay; this modification was made to accommodate the electrical current requirements of the heating strips. The controlling thermocouple was placed inside the processing tray and as close to the sample as possible; it was connected to both the data logger and the controller. During the inerting operations the temperatures of the sample (TC 1, Figure 1) and the atmosphere just inside the chamber walls (TC 2, Figure 1) was recorded by an Omega OM-160 data logger. The equipment configuration is shown in Figure 2.

The atmosphere within the chamber was controlled by an evacuation/purge system. The purge system consisted of a cylinder of ultra-high purity (UHP) nitrogen with a two stage regulator attached to the chamber via 1/4" stainless steel tubing. The evacuation system was driven by a 0.7 CFM, direct drive, rotary vane vacuum pump. On the vacuum side of the system was a four stage filtering train. The first stage was a 2 μ m, 25 mm diameter, glass fiber, in-line filter. This filter was to serve as a "witness" filter during the radioactive tests; it was to be a first stage collector for any radioactive particles generated during the tests. This filter was removed after the final test and was analyzed for radioactive contamination; it was found to be free of radioactivity. The second stage was a HEPA filter (MSA P/N 86684) to collect any particles that might be fine enough to pass through the witness filter. The third stage was a molecular sieve trap to stop moisture before it got to the vacuum pump; the final stage was the oil in the vacuum pump itself. Pump exhaust was into a fume hood.

Chamber Design Qualification

The chamber design is defined and documented in the drawing set already listed in this report. The adequacy of the design for these operations, from an explosives point of view, was verified by both calculations and testing. The chamber was sized to provide total containment of an explosion equivalent to 25 grams of Trinitrotoluene (TNT). The calculations took into account the estimated pressures which would act on both the cylinder walls and on the end closures. The longitudinal, hoop and radial stresses in the cylinder walls were calculated and found to be most severe for the hoop stress at ~ 13,647 psi. The circumferential and tangential stresses in the end closures were calculated and found to be most severe in the tangential mode at 11,250 psi. For the determination of safety factors, the material strength of 17-4PH stainless steel, condition H1025 at 350 °C was used. The yield strength of this material at 350 °C is ~ 130,000 psi. To be conservative, the estimated pressures were multiplied by a factor of two; to account

for the fact that the pressure load in this case is impulsive rather than static. The calculated factor of safety for this design is ~ 4.8 , i.e. the design could sustain 4.8 times the expected stresses. Therefore, the design was judged to be of adequate safety to meet the DOE's requirement that the system be "...capable of total containment of the effects of an explosion..."^{1b}, if the quantity of explosives was limited to < 25 grams of TNT or TNT equivalent.

In addition to the calculated safety factors, both DOE and SNL require that chambers used for explosives testing be tested to verify safe containment of $\geq 125\%$ of the stated explosive load limits of the chamber. Testing for this chamber was done in Area II, SNL/NM. The test used 32 grams of Hexanitrostilbene (HNS) explosive which has a 1/1 equivalency to TNT (see Table 2). Pre-test set-up evaluations indicated that a sample approximately centered in the cylinder could be brought to $300\text{ }^{\circ}\text{C}$ while the cylinder walls remained at $\sim 65\text{ }^{\circ}\text{C}$, this is a ΔT of $\sim 235\text{ }^{\circ}\text{C}$ with an air atmosphere in the cylinder. This test suggested that we would be able to heat samples to the required levels without compromising the properties of the cylinder. Based on those conclusions and due to the difficulties of doing a proof test at $> 350\text{ }^{\circ}\text{C}$, we elected to conduct the proof test at ambient temperature. Later discussions will show that even at the maximum temperatures achieved in these tests, the temperature of the chamber did not reach levels where the properties of the steel were significantly affected.

The results of that test are documented in Appendix A and summarized here. The dimensions of the chamber, including end closures and bolts, were measured pre- and post-test and found not to have been affected by the detonation. These results showed that the chamber was appropriately designed for containment of the effects of an explosion of 25 grams of TNT equivalency. However, for the 125% overtest there was some venting of the product gases. This result did not preclude the use of the cylinder, but did point out that, if the limits of the chamber were to be approached, the system should be used under an appropriate fume hood.

Inerting Procedure

The procedures for the inerting process are given in Appendix B. These are briefly described below. The procedures described below are for the treatment of the nonradioactive samples and may differ slightly from those in Appendix B, which are for the treatment of the radioactive samples.

The materials to be processed were pre-packaged prior to installation into the sample tray. In the case of detonators and igniters, where there was a probability of fragmentation if the units detonated or ignited, the units were placed inside a 6" long, heavy walled, 2"x 2" square, mild steel tube. The items were held in place in the tube by steel wool packing. In the case of the MC2370 firesets, which are mostly polycarbonate, they were wrapped in 2 layers of aluminum foil to form a pouch around the residue. The pelletized HNS explosive was also placed in a double layer of aluminum foil for residue containment.

The packaged materials were placed in the stainless steel tray and the tray was loaded into the chamber. During the installation, thermocouple #1 was inserted under the lid of the tray in close proximity to the sample being processed. The fiber glass insulating material was then

placed around the tray to insulate the tray from the chamber walls. The blank end closure was then bolted on with a new copper seal ring and the bolts torqued to 50 lb.-ft with a calibrated torque wrench. Prior to start of the heat cycle, the chamber was evacuated to > 20 " Hg vacuum. The chamber was then backfilled with nitrogen to atmospheric pressure and the evacuation to > 20 " Hg was repeated. This inerting and pump down was done in order to minimize the afterburn of any deflagration or detonation products that might occur during processing. Elimination of the afterburn was necessary to keep the pressure in the chamber as low as possible and to keep the non-electrical heating to a minimum. The temperature controller and the data logger were turned on immediately following the second pumpdown.

Once the temperature controller was turned on, and following a short induction period, the temperature rose at about $30\text{ }^{\circ}\text{C}/\text{min}$. Once the maximum temperature was reached, the process was allowed to continue for $\sim$ two hours of soak time, to ensure complete decomposition. After the soak time the controller was turned off and the system allowed to cool to ambient temperature. When ambient temperature was reached, the chamber again went through an evacuation and purge procedure identical to the one prior to beginning the heating cycle. This removed gaseous byproducts produced during decomposition. The chamber was then back filled with nitrogen to atmospheric pressure prior to opening. The decomposed materials were then removed for evaluation.

The data collected on the data logger was transferred to a laptop computer for analysis and plotting using the Omega/Pronto data analysis program provided with the data logger.

Discussion of Results

The process for treatment of the radioactive waste streams was developed by doing a series of inerting tests on components that were non-radioactive, but analogous to those in the radioactive wastes. These tests were carried out in building 940, Area II, SNL/NM. The actual radioactive inerting tests took place in the Hot Cell Facility (HCF), Tech Area V of SNL/NM. The temperature vs time data collected for those tests are given in Figures 3 - 5, for the analog tests and in Figures 6 - 7 for the radioactive tests. The data in Figures 3 - 7 are numbered corresponding to the information given in Tables 2 and 3 for each series of tests.

Temperature of the Chamber During Testing

Previously we indicated that, during proof testing of the inerting chamber the temperature of the chamber wall was considerably cooler than the temperature of the sample. Based on that observation we concluded that the material properties of the chamber would be negligibly affected by the bakeout cycle(s) that we intended to use. Figures 3C and 3D and Figures 6A and 6B show the difference in temperatures recorded by thermocouples 1 and 2 (see Figure 1) during two tests. Both of those tests were instances where the sample was taken to a mean temperature of just over $500\text{ }^{\circ}\text{C}$ and maintained for 58 minutes in one case and 107 minutes in the other. In both cases the ΔT between the mean temperature of the sample and the maximum temperature seen by the chamber wall was over $270\text{ }^{\circ}\text{C}$. Thus, it is safe to say that the safety margin of the chamber was not compromised at any time during these tests. Admittedly, there was one instance (Radioactive test # 4) when the recorded temperature from thermocouple 2

showed a maximum of 457 °C. However, when the chamber was disassembled for sample removal, it was discovered that the thermocouple had been inadvertently caught between the sample tray and the fiberglass insulation; thus, the measured temperature was not an accurate measure of the chamber condition and considerably closer to sample temperature than it should have been. In any case, these two examples serve to demonstrate that the samples can be taken to the levels required for inerting without compromising the integrity of the chamber.

Analog Tests

The temperatures used for the inerting of these materials runs were based on the data from Table 2 and the information from Table 3. For those samples that we thought had a good probability of producing fragmentation, i.e. those with confined explosives, we used a heavy walled tube to contain fragments. Those tests were numbers 5, 6, 7, 9, 10 and 11. This extra protection was used primarily to protect the sample tray from any unnecessary damage. Tests 7 and 11, which involved MC4217 detonators, did produce an audible signal similar to a popcorn sound when they detonated. The remaining tests did not produce any similar signals. With the exception of waste stream 910402-1, which contained explosives extruded into polycarbonate, a visual inspection was sufficient to establish that the explosives had been destroyed. Figure 8 shows a photograph of the post test MC4217s, the slight bulge in the middle of the detonator barrel indicates that the explosives detonated under confinement and were destroyed. The residual hardware for these and the other components tested showed thin, film like deposits of carbonized material on both internal and external surfaces, but no evidence of unreacted explosive.

In the case of sample 910402-1 simple visual verification of explosive destruction was not sufficient to establish the removal of the explosive. In this case the PETN portion of the extrudable explosive decomposed and left behind the silicone rubber matrix of the binder portion of the extrusion. The remaining silicone structure was suspended in a "foamed" polycarbonate matrix, shown in Figure 9. Analysis of the residual binder following one these test showed that most of the PETN had decomposed, but that a detectable, by high performance liquid chromatography (HPLC), trace of PETN was remaining. The remaining traces of PETN were removed by taking the "foamed" materials and reheating them in test #12. Following test #12 the "foamed" materials were completely charred, carbonized and lost the "foam" structure. The silicone binder material was still present, but HPLC analysis showed that it no longer contained any detectable PETN. Quantitative removal of PETN from a silicone rubber matrix has been described⁵ at temperature lower than the 500 °C that was used in test # 12. Thus, it was expected that the PETN in these samples would have been completely decomposed in the initial tests. Apparently the foaming of the polycarbonate provided sufficient insulation to the XTX8003 extrusion that it did not reach the maximum temperature recorded for the initial runs. Consequently test #12 was programmed to reach 500 °C; when the temperature reaches that level even the polycarbonate decomposes efficiently and provides no insulation to the explosive.

Figure 5B shows the temperature vs time data for test # 11. That test was to destroy simultaneously a one gram sample of HNS and two MC4217 detonators. Based on Table 2, the minimum bakeout temperature required for this test was 355 °C. The figure shows that the

sample was at or above approximately 300 °C for only 18 minutes, not the nearly two hour cycle time of the other tests. The reason for this apparently anomolous test was that the temperature controller was shut down due to a ground fault problem, due to a current leakage in one of the heater strips. In any case, when the chamber was opened after cooling all samples were found to be completely destroyed. The destruction of the detonators was expected, because we had heard the audible indication of detonation before the controller shut down. The destruction of the HNS was also expected because the temperature had gone sufficiently high to take the explosive beyond its exotherm level. The point of interest here is that when the HNS explosive was taken to its exotherm temperature, it was not necessary to continue heating for very long to insure destruction. None of this information is new or unexpected, but is cited here to show that when a large or bulky sample is taken to the appropriate temperature, it is not unreasonable to expect that the explosives have been destroyed. This is, in principle, the reason that thermal inerting is recommended for complex or threaded parts that may contain explosives.

As mentioned above under the description of the inerting procedure, a steel tube was used to provide containment of fragments for some of these components. For those tests which involved samples either too large to insert into the heavy walled tube, or which would not detonate, an aluminum foil "pouch" was used to contain the material. This pouch served to provide a convenient method for disposal of the residual materials after testing. Tests 3, 4, 8 and 12 involved unconfined explosives wrapped in aluminum foil, they produced no audible signal at all. In those tests it appears that the explosives simply deflagrated or decomposed rapidly in the oxygen starved atmosphere of the chamber. Figure 3B shows a spike in the temperature vs time plot. We believe that was due to the escape of the HNS decomposition gases from the foil pouch and their contact with the thermocouple. Figures 9 and 10 show the materials residual to some of those tests. Figure 9 is a picture of a destroyed polycarbonate block from an MC2370 fireset used in test #3. Figure 10 is a picture of the completely carbonized remains of an HNS pellet. The fact that a significant portion of the residue is still together, although completely carbonized, is proof that the material deflagrated instead of detonating.

Examination of the data in Figures 3 through 5 shows that, for the equipment and test setup used in this study, the ΔT s between maximum and minimum cycle temperatures are larger when the temperatures achieved are low. Based on that observation and due to the obvious need to take the polycarbonate containing samples to a higher temperature than anticipated, we decided to conduct all of the radioactive inerting tests at a temperature of 500 °C.

Radioactive Tests

The samples tested in this series of tests are described in Table 4 and the temperature vs time data for those tests are shown in Figures 6 and 7.

This series of tests consisted of six tests, four of which were nearly identical. Those four, nearly identical, tests were numbers 2, 4, 5 and 6 and involved the inerting of polycarbonate containing parts and pieces of MC2370 firesets. Those firesets were in a degraded condition

due to the testing that resulted in the induced radioactivity. The explosive content of those pieces was decreased from the listed amount due to that degradation. Since the explosives in these samples were considerably degraded already, it was decided that as many as four firesets could be treated at once. In the analog tests the firesets had been destroyed two at a time. With the exception of test six, all of those tests achieved a mean temperature of $\geq 500\text{ }^{\circ}\text{C}$ and produced a completely carbonized residual waste; identical in appearance to the waste from analog test #12, which was also taken to $> 500\text{ }^{\circ}\text{C}$.

In test # 6 the sample tray only reached a maximum of $435\text{ }^{\circ}\text{C}$, but was above the minimum bakeout temperature for the explosive, Table 2, for ~ 110 minutes. When the sample for this test was removed for visual inspection, the samples were in the same carbonized condition as the samples from tests 2, 4 and 5 and samples from analog test #12, discussed above. We think that the reason the temperature did not rise as fast as in the other tests was that the heating strips may have begun to degrade and/or lost intimate contact with the sample tray; the same type of behavior can be seen in the early stages of test #4, but in that case the problem was not as severe and the sample did achieve a cyclic heating level.

Test #1 consisted of a variety of components including detonators and pyrotechnic, igniter devices. The MC4217 detonators listed in Table 4 were dissected units and did not provide confinement of the explosives to the degree of the analog units tested earlier. For that reason, those detonators were not expected to detonate in the manner of the units tested earlier. Post-test inspection of the residual waste stream showed that the explosives had decomposed; but no audible report had been heard as had been the case in the analog tests. The various units were mixed for this test because analog test # 10 had shown that a variety of types could be decontaminated simultaneously. The residual hardware showed the effects of the heating and looked very similar to components tested in the analog series, i.e. they were discolored and covered with a thin film of carbon. Photographs were not taken because the materials were in a radioactive material management area (RMMA) and any camera, or other equipment, that went into the area would be considered contaminated and have to be certified free of radioactivity before it could be removed from the area. Since we determined that the appearance was very similar to the non-radioactive residuals, we decided not to go to the expense of doing swipes and analysis of cameras, film etc. for these tests.

Test # 3 involved the destruction of HNS explosive, thus the temperature required, per Table 2, was $355\text{ }^{\circ}\text{C}$. Figure 6D shows that a mean temperature of $\sim 499\text{ }^{\circ}\text{C}$ was achieved. This is important because the explosive in this sample was distributed over several paper tissues and was in a conductive plastic bag. For this test the controlling thermocouple was inserted into the bundle of tissue, as close to center of the bundle as possible. This arrangement assured that the explosive reached the required temperature and was destroyed. In addition, some of the tissue in the bundle seemed to be still very lightly damp, probably due to the use of some water during the clean up operation, and served to conduct the heat throughout the bundle. The presence of this moisture turned out to be beneficial for another reason. After the test series was complete the witness filter was removed and submitted for analysis for ^3H contamination; it was found to be free of activity. When removed, the filter was found to be very damp and

would have formed a good exchange medium for ^3H from the HNS. The lack of any indication of contamination suggests that the explosive was not contaminated.

Post-Test Survey of the Equipment

Following the final test of the Radioactive test series our sponsor requested that the chamber, vacuum pump, data logger, controller and other equipment be packaged for delivery to their facilities for storage or use on another task. This required, of course, that all the equipment be surveyed for contamination prior to packaging. For this survey the equipment was disassembled and laid out for the analysts from Industrial Hygiene and the witness filter was removed and packaged for analysis. Swipes taken from all the equipment, including the inside of the chamber itself, were found to be negative for the presence of any radioactive contamination.

Conclusions and Recommendations

The treatability study that was described in this report shows that, for small quantities of radioactive explosives and/or small explosive components, an explosion proof chamber will serve well as a inerting chamber. There are, however, several points that can be made regarding alternate or better methods for using the chamber that might enhance its applicability. Those recommendations are made below.

The sample tray that was used in this study was constructed in the laboratory from commercial, laboratory hardware. As such, it was not ideal for all the applications, thus the use of the heavy walled tube for treating the detonators. We recommend that a heavy or heavier walled tray should be designed for this work. The tray should provide a mounting feature for the heating strips, or possibly for cartridge heaters, and a well for the thermocouple. These features would facilitate the manipulation of both the tray and the sample.

The heating strips that we selected for this study are rated for a maximum temperature of 600 °C and they, for the most part, worked well. But, we did appear to have problems with them after several runs. Admittedly, we are not certain whether the problems were due to degradation of the strips or to wear on the mounting mechanism; the point to be made here is that, thinking in retrospect, better mechanisms are available for supplying the heat. Half cylinder heating configurations could be used, for example.

And finally, we used a simple on/off temperature controller and a data logger for our tests. These components were used primarily as a cost reduction measure in the event that we did experience some contamination of the equipment during the tests. The cost of the data logger and the controller would be less than the cost of a digital recorder with input/output ports, etc. After this study we found that the probability of contamination from treating the waste streams of concern is probably quite low. Thus, we would recommend that in future efforts a more automated system be considered. The use of a proportional temperature controller would also minimize the ΔT seen in the long periods of cycling used to ensure complete inerting.

Table 1. Description of Waste Streams

RMW #	Description of Contents	# Units	N.E.W.
930192-1	One fragmented pellet of HNS, laboratory tissue used to clean up pellet fragments in the laboratory. Contains paper, plastic tape and Velostat plastic materials.	1 pellet	≈ 2 gm HNS
910402-1	Remains of dissected explosive components(MC2370 firesets, 2.46 gm PETN ea.) irradiated at Brookhaven National Labs. Contain PETN, XTX8003(80/20 PETN/Sylgard), polycarbonate, ceramic and electronic components and solder.	13 ea.	32 gm PETN
940025	Laser diode igniters loaded w/ Titanium Potassium Perchlorate (Ti/KP), 12 mgm ea. Activated in underground test. Contain stainless steel, ceramics, fiber optic and insulation.	4 ea.	48 mg Ti/KP
940026	MC4217 detonators loaded w/PETN, 28 mg ea. Activated in underground test and evaluated post-mortem. Contain stainless steel, plastics and glass ceramic.	10 ea.	280 mg PETN
New Waste*	Laser diode detonators loaded w/ CP with 1% carbon black(CB), 20 mgm ea. Activated in underground test. Contain stainless steel, ceramic, fiber optic and insulation.	3 ea.	60 mg CP/CB

* This stream was new and had not yet been assigned a unique number.

Table 2. Thermal Characteristics and Inerting Requirements of Some Explosives.^a

Explosive	Melting Pt.(°C)	Exotherm(°C)	Bakeout Temp.(°C)	%TNT Equivalence
PETN	141	150	210	174
HMX	280	235	295	160
HNS	318	295	355	100
TNT	81	260	320	100
CP	--- ^b	240	300	Unk.
TiKP	--- ^b	430	490	N/A
TiH _x KP	--- ^b	370	450	N/A

a. The data in this Table was compiled from references 2,3 or 4 or from in-house data.

b. These materials do not melt, they decompose rapidly, either by deflagration or detonation, on reaching their exotherm temperature.

Table 3. Description of Tests Performed on Non-Radioactive Analog Materials.

Analog Test #	Components Tested	T _{min} (°C)	T _{max} (°C)	T _{mean} (°C)	Time ≥ T _{min}
3	MC2370 Fireset (explosive lens only, ≈2.4 gm PETN), 1 ea.	209.6	314.0	254.1	165.3
4	HNS Pellet, 1 gm	290.2	434.0	337.9	129.2
5	Titanium/Potassium Perchlorate(12 mgm Ti/KP w/1% CB) pressed into charge holder, 3 ea.	473.2	531.0	501.8	57.8
6	MAD1174s (37 mgm CP w/1% CB), 3 ea.	232.7	380.0	280.3	163.0
7	MC4217 detonators (21.7 mgm CP/9.2 mgm HMX), 2 ea.	295.9	390.0	322.9	104.3
8	MC2370 Firesets (explosive lens only, ≈2.4 gm PETN), 2 ea.	210.8	403.0	254.4	171.0
9	MC2427 detonators (58 mgm PETN), 3 ea. From disassembled MC2370s.	210.8	392.0	270.0	109.1
10	MAD1174s, 4 ea.; MAD1186s (35 mgm CP w/1% CB), 7 ea.; Charge holders (CP w/5% CB), 7 ea.; Charge holders (CP w/1% CB), 2 ea.; Ti/KP Charge holders, 2 ea.	489.3	520.0	505.3	158.3
11	HNS pellet(1 gm), 1 ea.; MC4217s, 2 ea.	299.7	387.0	N/A	18.2
12	Retest MC2370s (residuals from tests 3 & 8).	487.5	517.0	502.0	142.8

Notes: 1) Time above T_{min} is in minutes, see Figure 3A.

2) See Figure 3A for definition of T_{max} & T_{min}.

Table 4. Description of Inerting Tests for Radioactive Components.

Rad. Test #	Materials Tested	T_{min}(°C)	T_{max}(°C)	T_{mean}(°C)	Time ≥ T_{min}
1	MC4217s(CP/HMX), dissected, 10 ea.; Laser diode igniters(Ti/KP), 4 ea.; Laser diode detonators, (CP/CB), 5 ea.	495.9	509.0	504.1	106.7
2	MC2370 Firesets, 3 ea., heavily damaged, partially decomposed and dissected. Also, tissue wrapping materials and some yellow tape.	495.8	509.0	503.3	86.2
3	HNS, ≈2 gm in Velostat bag, moist lab tissue used for explosives cleanup operations.	477.9	516.0	498.9	95.4
4	MC2370 Firesets, 4 ea., heavily damaged, partially decomposed and dissected. Also, tissue wrapping materials and some yellow tape.	489.8	511.0	500.8	78.4
5	MC2370 Firesets, 3 ea., heavily damaged, partially decomposed and dissected. Also, tissue wrapping materials and some yellow tape.	488.9	513.0	503.0	110.4
6	MC2370 Firesets, 3 ea., heavily damaged, partially decomposed and dissected. Also, tissue wrapping materials and some yellow tape.	211.0	435.0	N/A	109.7

Note: Time above T_{min} is in minutes.

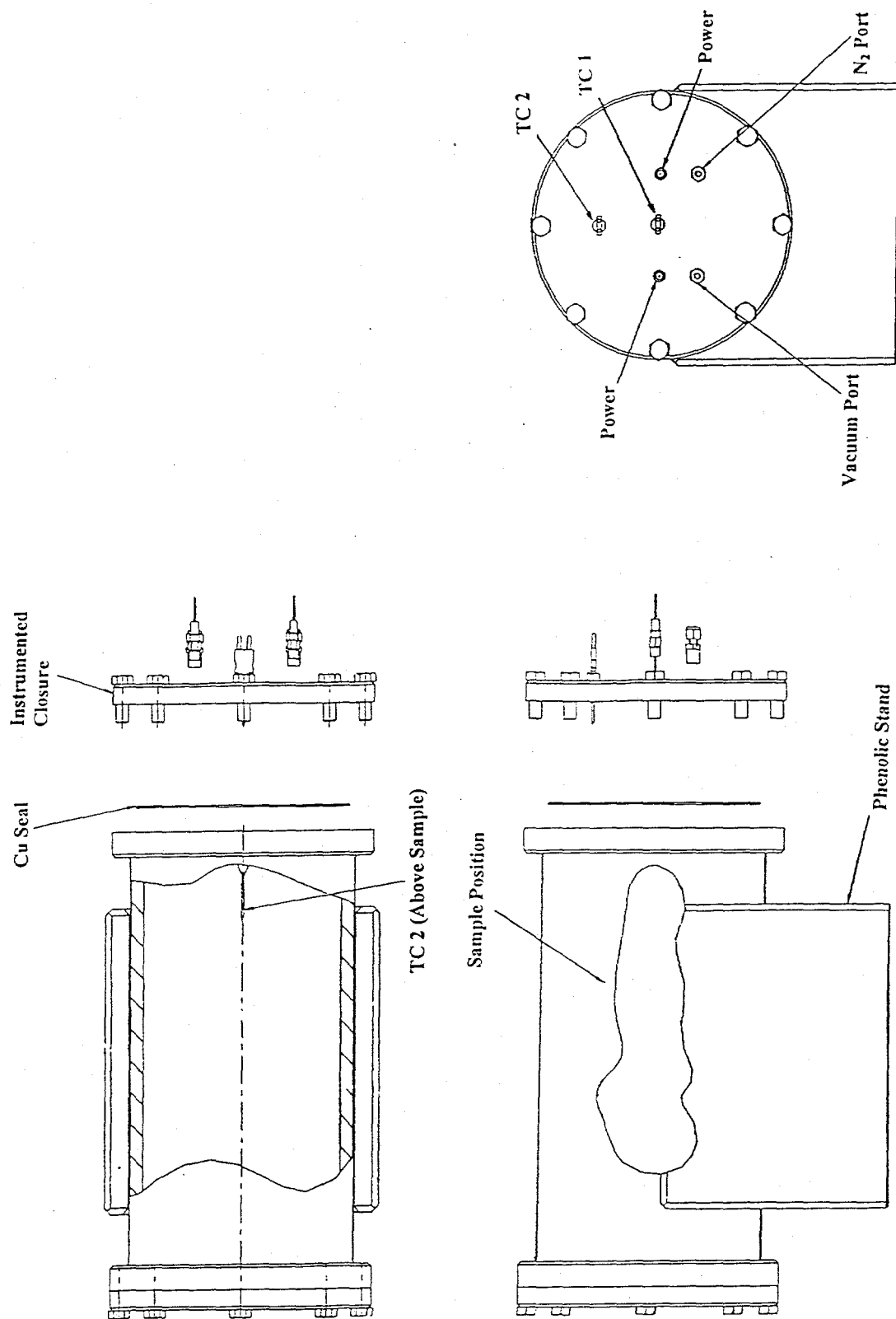


Figure 1. Schematic Figure of Inerting Chamber.

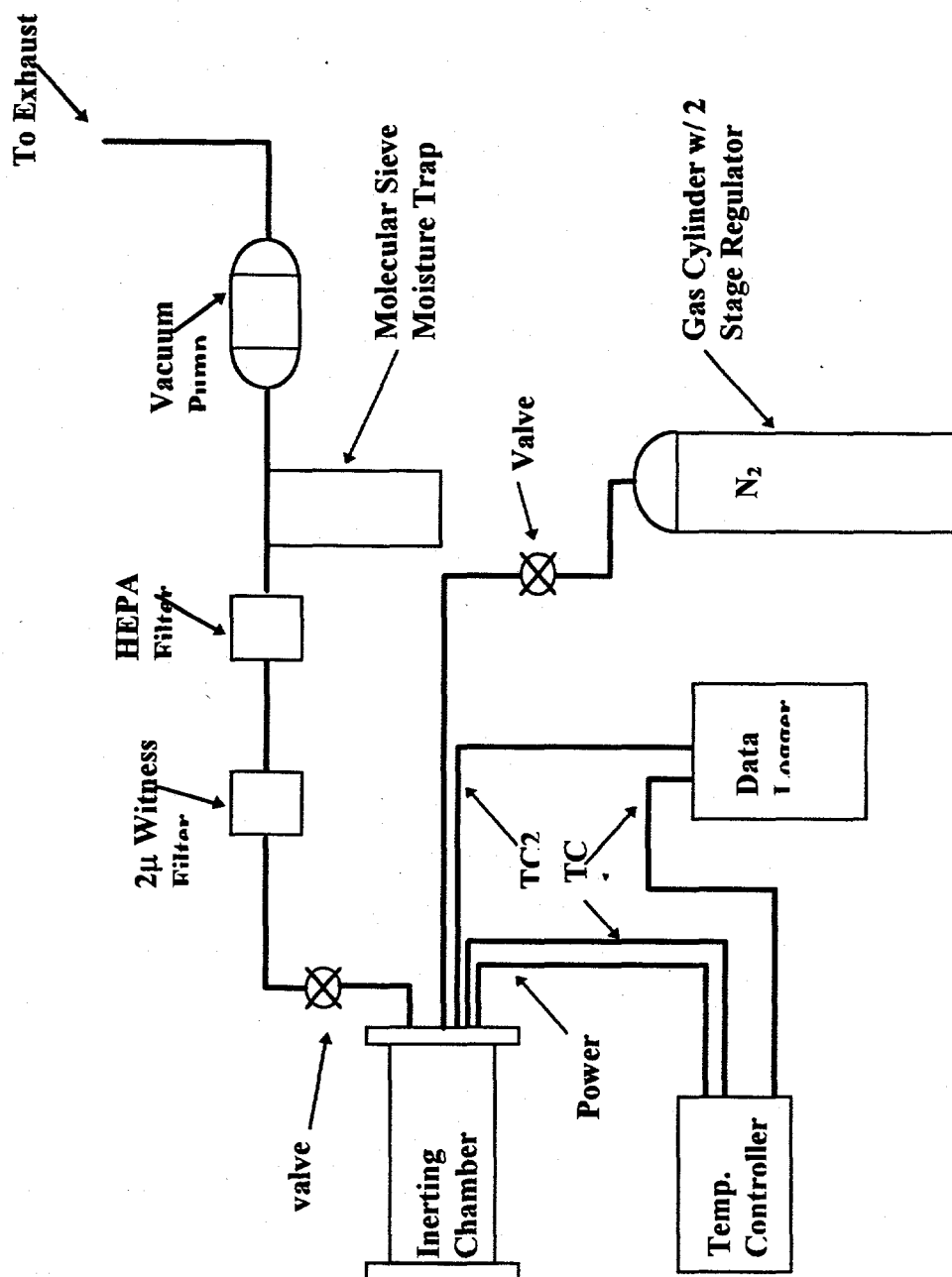
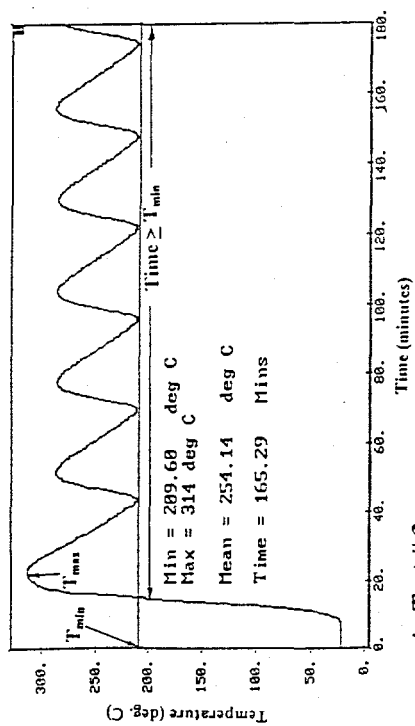
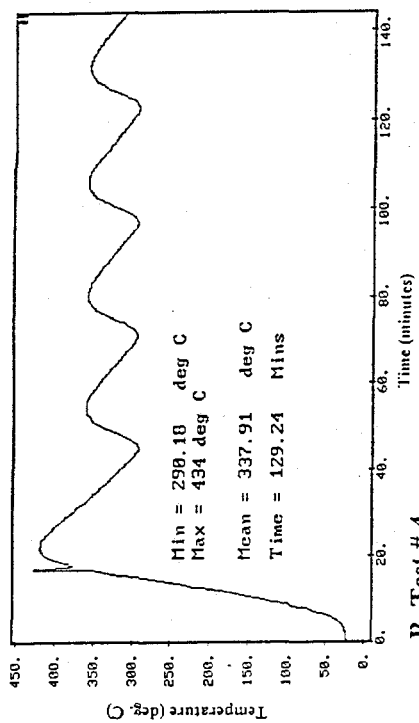


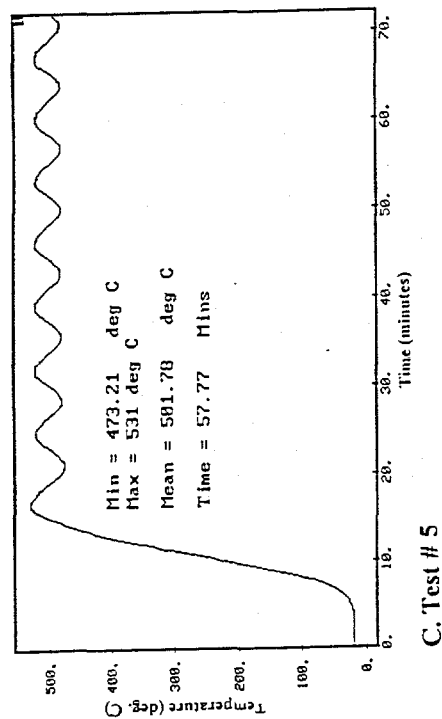
Figure 2. Schematic Layout of Thermal Inerting System and Ancillary Equipment.



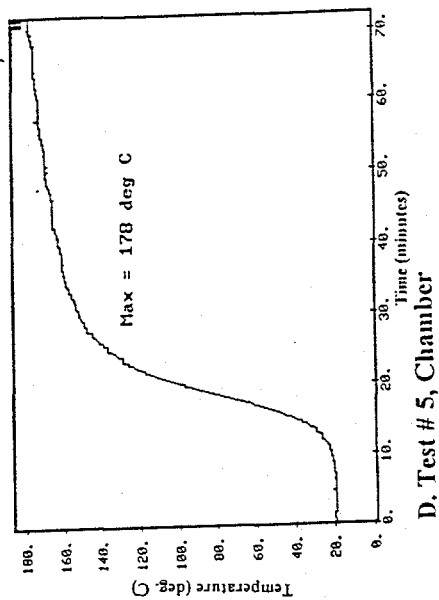
A. Test # 3



B. Test # 4



C. Test # 5



D. Test # 5, Chamber

Figure 3. Temperature vs Time; Analog Tests 3, 4, and 5.

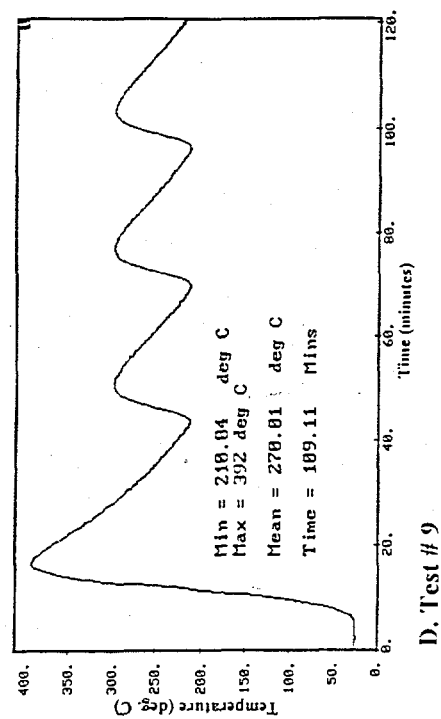
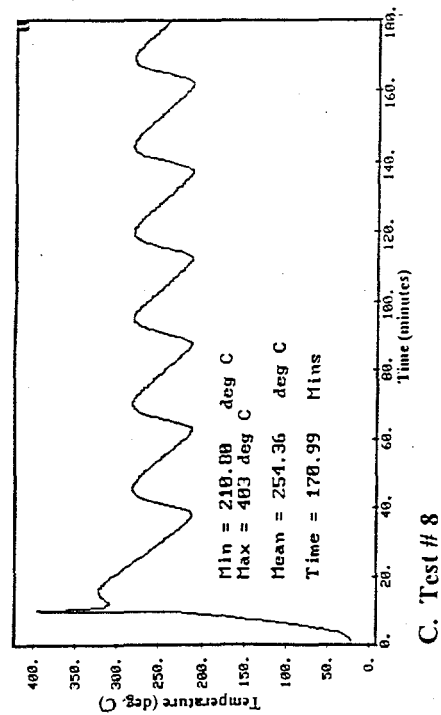
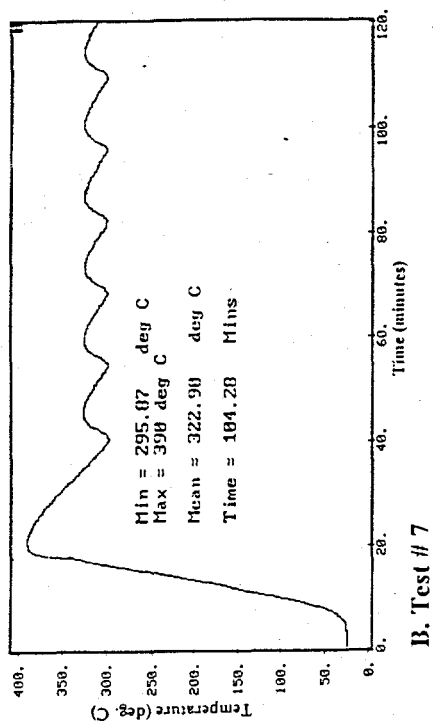
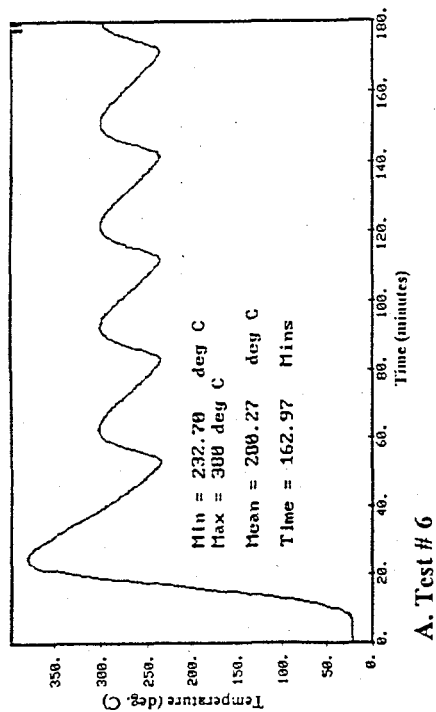
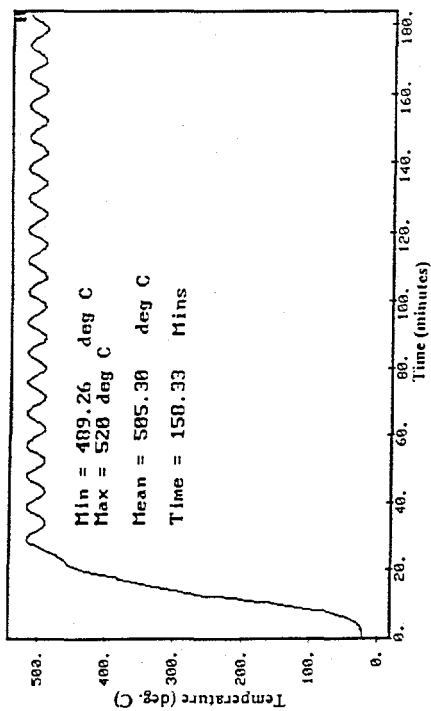
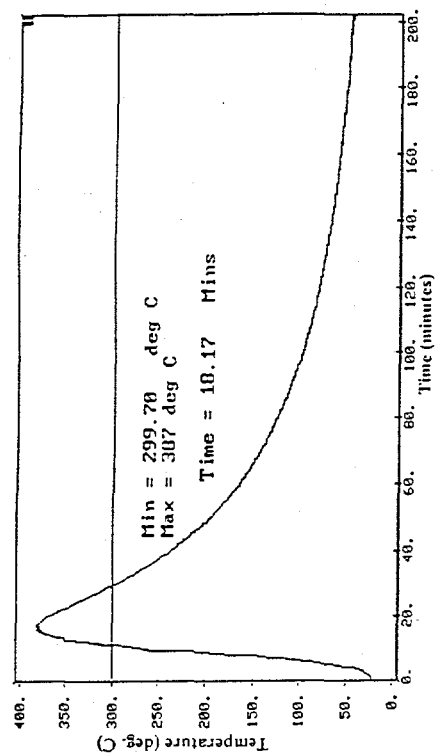


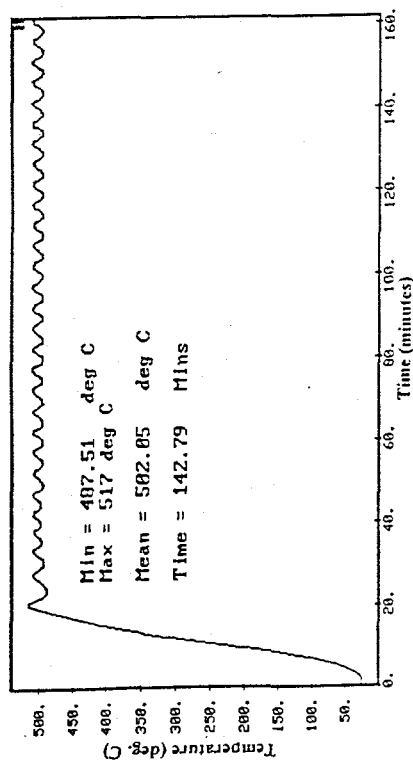
Figure 4. Temperature vs Time; Analog Tests 6, 7, 8 and 9.



A. Test #10



B. Test #11



C. Test #12

Figure 5. Temperature vs Time; Analog Tests 10, 11 and 12.

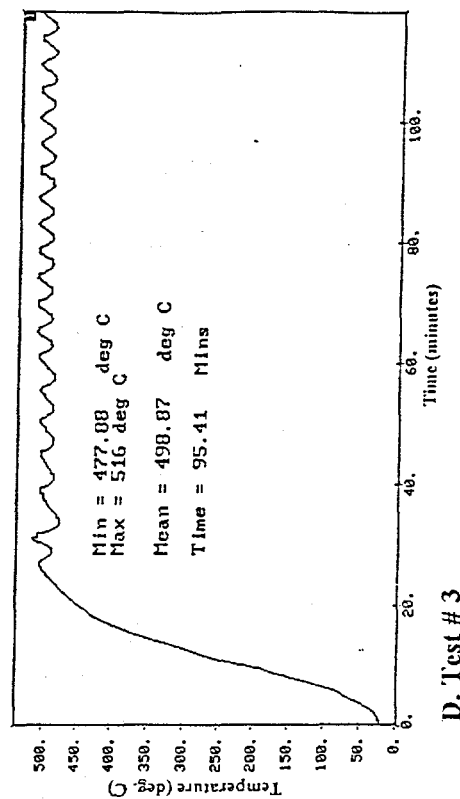
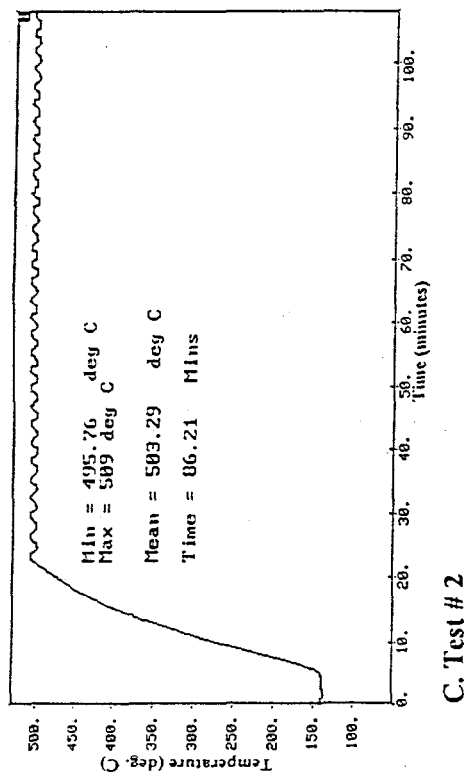
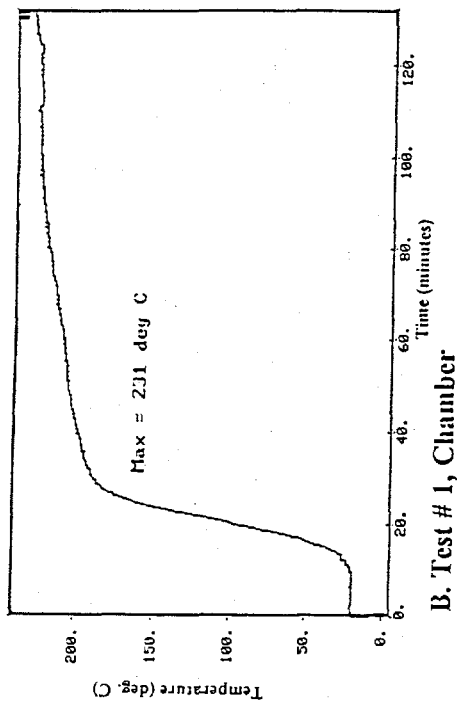
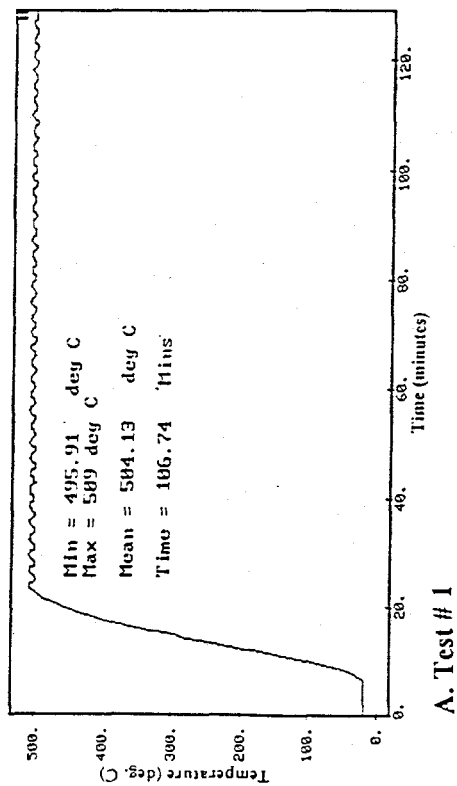
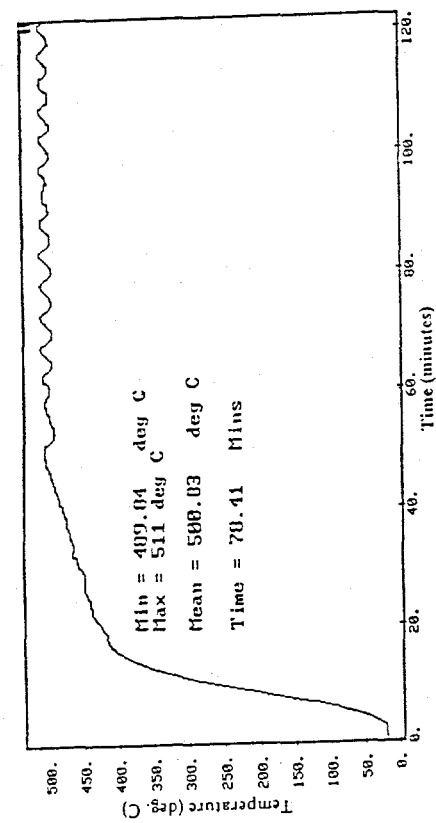
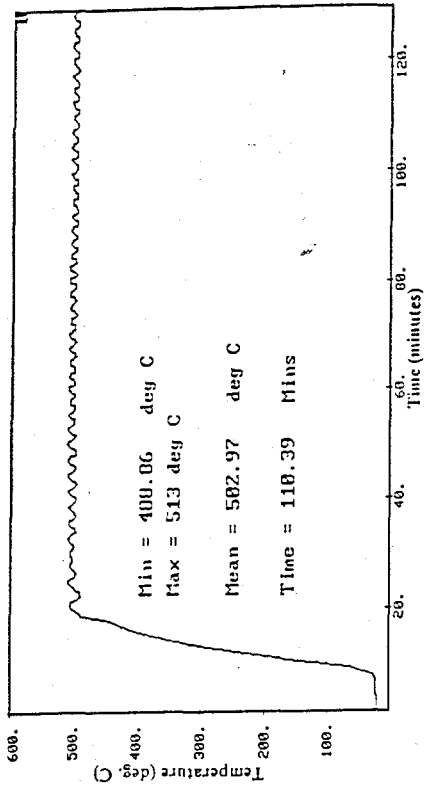


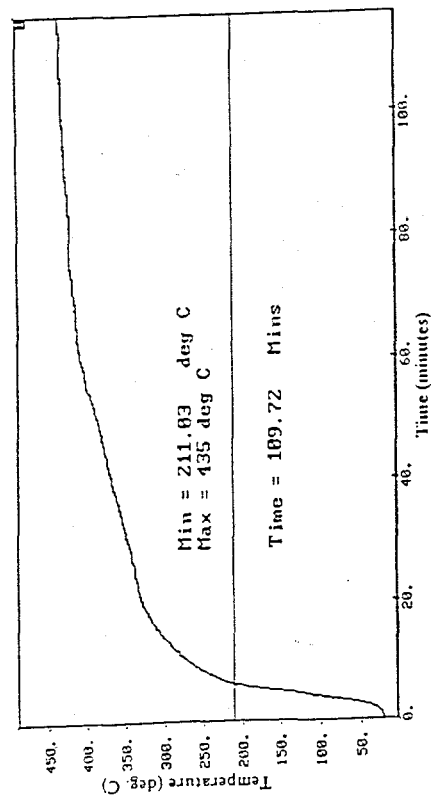
Figure 6. Temperature vs Time; Radioactive Tests 1,2 and 3.



A. Test # 4

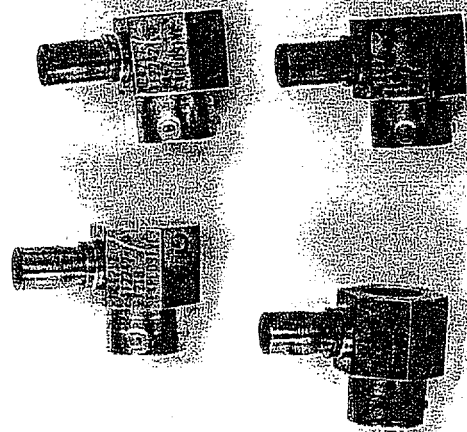


B. Test # 5



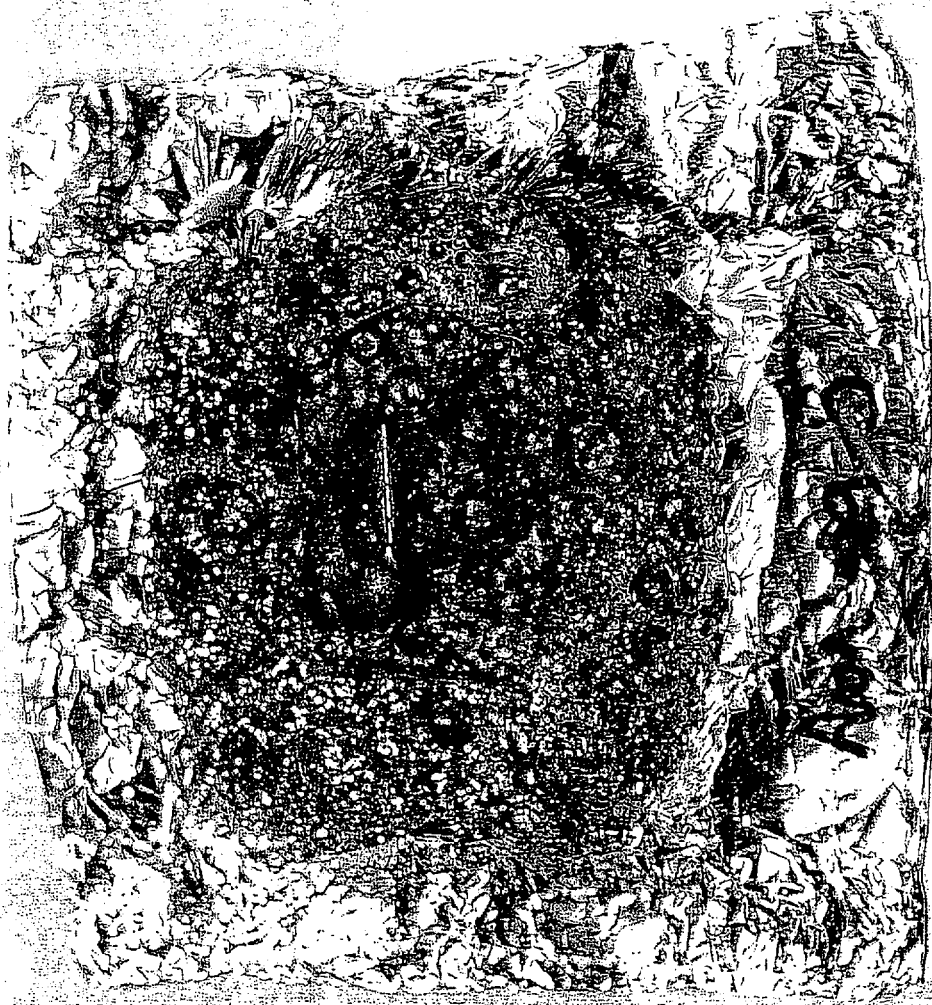
C. Test # 6

Figure 7. Temperature vs Time; Radioactive Tests 4, 5 and 6.



MC4217 Detonators

Figure 8. Thermally Destroyed MC4217 Detonators.



MC2370, Fireset

Figure 9. Foamed Polycarbonate Block From MC2370.

HNS Pellet



Figure 10. Charred Residual Materials From Decomposition of HNS.

References

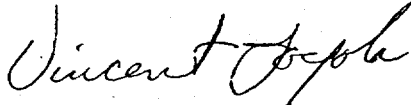
1. DOE Explosives Safety Manual, DOE/EV/06194, Revision 7.
 - a) Chapter II, Section 18, "Decontamination and Cleaning"
 - b) Chapter II, Section 21, "Laboratory Operations"
2. LANL Explosives Handbook.
3. U.S. Army Material Command Pamphlet, AMCP 706-177, January 1971.
4. Explosives, 3rd Ed., Rudolf Meyer, VCH, 1987.
5. LX-13/PEEK Compatibility Study Interim Report; Patricia A. Foster, Mason & Hanger - Silas Mason Co., Inc., Pantex Plant; William Andrzejewski and Jean Womelsduff, Sandia National Laboratories, Albuquerque, NM; December, 1986.

Appendix A

SANDIA NATIONAL LABORATORIES
Albuquerque, New Mexico 87185

Date: March 7, 1995

To: J. E. Dotts, 7732, MS 1045



From: Vincent Loyola, 2652, MS 0329

Subject: Proof Test of Cylinder Assembly, R45632.

Attached is a short report on the results of proof testing of stainless steel cylinder assembly R45632. This cylinder assembly will be used in a project to inert some energetic materials and/or components in Area II. The Area II tests will be in preparation, i.e. process prove-in tests, for inerting similar components in Area III. The operations in Area III will involve some materials which have been activated to low level radioactivities. The Area III work is part of an effort to determine if the explosive hazard can be safely and cleanly removed from radioactive explosive components. The intent is to demonstrate this approach for reducing explosive mixed waste to non-explosive mixed waste.

The test setup for this proof test was reviewed by Vern Hermansen prior to the test. Vern was asked to review the test setup because you were out of your office when the test was performed.

Please review the report and let me know if you agree with the conclusions. If you do not agree, please let me know what needs to be done to qualify this steel vessel for use.

Copy To:

MS 0329	J. G. Harlan
MS 0329	V. M. Loyola
MS 0329	S. D. Reber
MS 0327	R. V. Saxton
MS 0326	G. L. MacCosbe
MS 1045	V. Hermansen
MS 1303	P. K. Peterson

March 7, 1995

Introduction:

This report describes the proof test that was performed on a stainless steel cylinder (P/N R45632) that was designed for the safe thermal decomposition of small quantities of explosives and/or explosive components. The cylinder will be used to remove the explosive hazard from small quantities of explosive mixed waste. At the present time there is apparently no mechanism for the management of explosive contaminated mixed waste. As a consequence, organizations that have such waste in their possession have no alternative but to continue to store it in their areas. The removal of the explosive hazard from these waste streams will enable the mixed-waste management organization to take possession of the waste and process it per accepted methods.

These waste streams were generated by various organizations during radiation testing of materials and components. Generally, these waste streams consist of small quantities of explosive materials configured as either loose powders, pressed powders or as packaged components. At the time that these waste streams were generated there were few or no restrictions on the generation or disposal of such wastes; in today's climate, the production of such wastes is severely restricted and in some cases prohibited. However, in the event that generation of such waste in the future is unavoidable; we hope that the methods developed in this effort will be useful in avoiding the dilemma in which most of the generators of the past now find themselves.

Methodology:

The method(s) to be developed in this project for the inerting of energetic materials will be thermal methods. In the context of this work "thermal method" means a mechanism by which a decomposition reaction, deflagration and/or detonation can be induced in the material by application of a thermal stimulus. The intent is to remove the reactive nature of the materials while minimizing, to the extent possible, the production of additional radioactive waste and avoiding the production of liquid effluent. In order to achieve those goals, the approach to be taken will be to bake-out the energetic materials under temperatures sufficiently high to achieve irreversible destruction of the energetic nature of the materials.

The materials with which we are presently concerned include the high explosives PETN, HNS and CP as well as some pyrotechnic material, Titanium/Potassium Perchlorate (TiKP). However, the mechanisms developed will be applicable to any energetic material which can be induced to undergo a thermal decomposition. Other materials which we may encounter are shown in Table 1 below, the highest temperature to which we expect to take any high explosive material or component is 355° C, i.e. for HNS or HNS loaded components. The pyrotechnic materials will require a much higher temperature, but, since these materials do not detonate, the explosive hazard associated with inerting these materials in our device is much reduced and practically non-existent.

March 7, 1995

Cylinder Design and Test:

The definition of the inerting cylinder is contained in the following drawings:

R45632 ; Assembly, Heating Cylinder
R45634 ; Closure, Cylinder
R45659 ; Bolt, Cylinder
R45660 ; Support, Cylinder

R45633 ; Cylinder
R45652 ; Closure, Cylinder
R45635 ; Assembly, Heating Plate

Briefly, the overall design is a stainless steel cylinder of 0.5" wall thickness and 8.0" inside diameter. The end closures are stainless steel and 0.8" thick; one of the end closures has threaded ports for the installation of power feedthroughs for the heater(s), for thermocouple fittings for the temperature controller, and for compression fittings to permit the evacuation and backfilling of the internal volume before and after inerting. The cylinder is sealed during operations by the use of a copper gasket and knife edge seal.

The cylinder was designed to contain, without damage and deformation, an equivalent of 25 grams of TNT at a temperature of 350° C and was tested with 32 grams of HNS, a 125% overtest. The overtest, however, was done at ambient temperature; the reason for this is discussed below. The material of construction is 17-4PH stainless steel at a heat treat condition of H1025. This material is a high temperature steel designed for applications requiring corrosion resistance and high strength at temperatures up to 316° C. The properties of the material at 350° C were used for the design calculations. The properties at 350° were used because it is close to the highest temperature that we anticipate using for inerting explosives which are common to SNL explosive devices, see Table 1 below. Theoretical calculations were done to determine the expected pressures due to the detonation of 25 grams of TNT. The maximum stresses expected in the cylinder and in the closure bolts were calculated based on those predicted pressures. The lowest safety factor thus determined was almost three at 350° C; that number was for the closure bolts, all other factors were five or higher.

As indicated above, the design calculations assumed a temperature of 350° C but the test was done at ambient. The reason for this was that I feel that the configuration of the hardware during the inerting tests will preclude the cylinder ever approaching that temperature level. Testing of the empty cylinder prior to doing the overtest showed that when operated as intended in the governing OP (OP471574), the test specimen, located approximately centered in the cylinder, could reach ~300° C while the cylinder itself reached a temperature of only ~65° C, a ΔT of 235°. This suggests that under operating conditions the explosives can be thermally decomposed at well above 350° C while the cylinder will remain well below a temperature level where its mechanical properties are effected. Based on that empty cylinder test, I concluded that an overtest at ambient conditions would give results which are valid at the prescribed operating conditions. A copy of a plot of Tensile/Yield Strength vs Temperature for this material is attached.

Test Results:

The test showed that the cylinder design and construction will provide the safety required for explosives operations as described in OP471574.

To: J. E. Dotts, 7732
Subject: Proof Test of Cylinder

March 7, 1995

The pre- and post test dimensions of the cylinder are shown in Table 2. These data show that the cylinder withstood the detonation of the 32 grams of HNS explosive with no damage and/or measurable deformation. The closure bolts, which have the lowest calculated margin of safety, were completely unaffected.

The only problem that we found was that the copper gasket seal did leak some of the detonation gases. I have what I believe is a reasonable explanation for this observation, and a possible solution. The copper gasket/knife edge seal works based on the metal surface to metal surface contact between the soft copper and hard steel; this contact is maintained in vacuum applications by the ΔP load on the closure. In the case of our cylinder, this load was applied by the torque on the bolts, which were torqued to 30 ft-lbs. I believe that when the detonation occurred the shock waves produced by the detonation passed through the cylinder walls and the closures and were coupled or transferred from one part to the other through copper gasket. The passage of the shock waves through the copper resulted in two different things, both of which caused a failure of the seal. First, the passage of the shock wave(s) broke the metal to metal contact between the soft copper and the hard steel. Second, the reflected shocks caused the closure and the cylinder to move closer together under an "instantaneous" load thus causing additional compression of the gasket and loss of torque load on the bolts. Either or both of those effects would cause the gasket to lose its seal.

The solution to that problem will be to apply sufficient torque to the closures to bring the cylinder and closure surfaces into metal to metal contact, thus reducing the ability of the parts to re-compress the gasket. This approach, coupled with the expectation that explosive loads under actual use will be no more than 10 grams at a time and in a configuration that is not expected to detonate, only deflagrate or decompose, should dramatically reduce any shock loading to the gasket.

It is my conclusion that the cylinder, as designed and for its intended operations, is adequately safe to permit its use as proposed in OP471574.

March 7, 1995

Table 1
Critical Temperatures of Explosives
for Use With This OP

The conditions to which the explosive materials will be subjected during inerting operations described in this OP will be very similar to conditions existent in a Differential Scanning Calorimeter (DSC). Consequently, under the conditions of those operations and consistent with the definition of T_c , the critical temperature (T_c) of the explosives will be considered to be the same as the "Onset of Exotherm" temperature measured by DSC.

Unless otherwise indicated, the % TNT values for these explosives have been taken from Explosives, 3rd Ed., Rudolf Meyer, VCH, 1987.

<u>Explosive</u>	<u>Atomic Composition</u>	<u>Mol. Wt. (gm/mole)</u>	<u>DSC Melt. Pt. (Deg. C)</u>	<u>Minimum Exotherm* Temp. (C)</u>	<u>Bakeout Temp. (C)</u>	<u>% TNT Trauzel</u>
PETN	$C_5H_8N_4O_{12}$	316.1	141	150	210	174
β -HMX	$C_4H_8N_8O_8$	296.2	280	235	295	160
RDX	$C_3H_6N_6O_6$	222.1	204	200	260	160
HNAB	$C_{12}H_4N_8O_{12}$	452.2	221	265	325	
HNS	$C_{14}H_6N_6O_{12}$	450.1	318 ^b	295	355	100
TNT	$C_7H_5N_3O_6$	227.1	81	260	320	100
CP	$C_2H_5N_{10}O_8Cl_2Co$	437.0	Dec. ^c	240	300	
TiKP	Ti/KClO ₄	Blend	Dec. ^d	430	490	N/A
TiH _x KP	TiH _{1.65} /KClO ₄	Blend	Dec. ^d	370	450	N/A

Notes:

- a: These data represent the temperature at which these materials begin a self-sustaining, irreversible decomposition, either deflagration or detonation.
- b: HNS undergoes decomposition as it melts: Rudolf Meyer, Explosives, 3rd Ed., VCH, 1987.
- c: CP does not melt, it decomposes violently at the given temperature.
- d: TiKP and TiH_xKP, in its various H_x forms, do not melt, they auto-ignite and deflagrate rapidly when heated to the given temperature.

To: J. E. Dotts, 7732
Subject: Proof Test of Cylinder

March 7, 1995

Table 2
**Pre- and Post Test Dimensions of
Cylinder Assembly, P/N R45632**

Test Parameters:

Temp.: Ambient TNT Eq.: 32.1 gm (31.24 gm HNS @ 100 % TNT + .63 gm PETN @ 150 % TNT, RP-1)
Initial Torque on Bolts: 30 ft-lbs

Cylinder Dimensions:

<u>Pre-Test</u>	<u>Position</u>	<u>Post Test</u>
7.999"	# 1 Diameter	8.000"
7.999"	# 2 Diameter	7.999"
18.081"	Inside Length	18.082
.001" Uniform	Closure Plates	.001" Uniform
Conical, center thickness	Both Ends	Conical, center Thinness

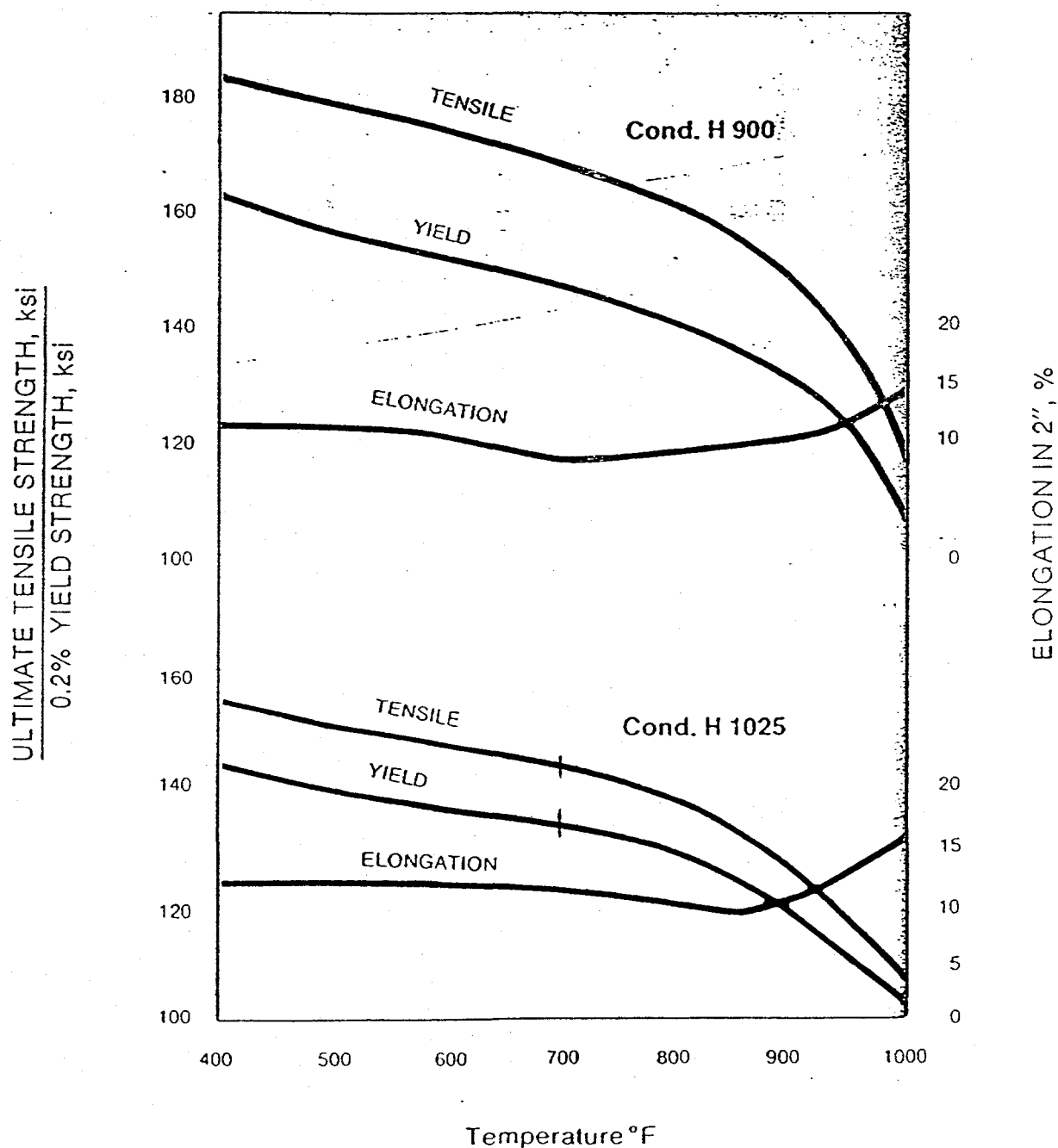
Bolts:

<u>Bolt #</u>	<u>Pre-Test</u>	<u>Post Test</u>
1	1.820"	1.820"
2	1.820	1.823
3	1.820	1.821
4	1.823	1.824
5	1.818	1.819
6	1.822	1.821
7	1.822	1.822
8	1.819	1.819
9	1.823	1.821
10	1.822	1.822
11	1.820	1.820
12	1.819	1.819
13	1.822	1.822
14	1.822	1.822
15	1.825	1.825
16	1.825	1.826

Typical Short Time Elevated
Temperature Properties
of Republic
17-4 PH and 15-5 PH

Condition H 900
and H 1025

Longitudinal Mechanical Properties



Sandia National Laboratories

Albuquerque, New Mexico 87185-1045

date: March 16, 1995

to: Vincent Loyola, MS-0329 (2652)



from: J. E. Dotts, MS-1045 (7732)

subject: Proof Test of Cylinder Assembly, R456312

I have reviewed the report on the results of proof testing of the subject assembly and I concur with your conclusions.

Copy to:

MS 1045 V. L. Hermansen, 7732

MS 1045 J. E. Dotts, 7732

MS 1045 File, 7732

Appendix B

TABLE OF CONTENTS

1.0 PURPOSE, SCOPE AND OWNERSHIP	3
1.1. Purpose	3
1.2 Scope 3	
1.3 Ownership	3
2.0 RESPONSIBILITIES	3
3.0 TRAINING QUALIFICATIONS	3
4.0 DEFINITIONS	4
5.0 HAZARDS	5
6.0 SAFETY EQUIPMENT	6
7.0 STANDARD OPERATING PROCEDURE	6
8.0 WASTE DISPOSAL	11
9.0 ES&H REPORTING AND DOCUMENTATION	11
10.0 REFERENCES	12
11.0 APPENDICES	12
12.0 AUTHORIZED USERS LIST	13

1.0 PURPOSE, SCOPE AND OWNERSHIP

1.1. Purpose

This OP describes how explosives and small explosive components which are radioactive due to exposure to ionizing radiation will be handled before, during and after inerting of the energetic material.

1.2 Scope

This OP applies to Department 2552 personnel or other personnel who will be involved with the inerting of small quantities of radioactive explosives and explosive components in the Hot Cell Facility (HCF), Building 6588, located in Tech Area V.

1.3 Ownership

This OP is jointly owned by Explosive Subsystems and Materials Department (2552) which will perform the required tasks and Nuclear Facilities and Diagnostics Department (14621) which has ownership of the site listed above. Comments and concerns about this document or any ideas for improvement should be sent to the author or either one of the Department Managers.

2.0 RESPONSIBILITIES

Department 2552 personnel are responsible for performing the activities described in Section 4 of this OP. Personnel who will be involved in these activities will read and sign this OP, 6521-HCF-RCM-01 and SOP SP473319. The 2552 personnel performing the tasks in Section 4 of this OP are responsible for maintaining the equipment required in Section 4 of this OP. The HCF facility supervisor is responsible for maintaining the PHA for the facility and for determining if additional training is required for operation in the HCF facility.

3.0 TRAINING QUALIFICATIONS

3.1 ES&H Training Requirements

All SNL employees and onsite contractors who work under the guidelines of this OP are required to be in compliance with the training requirements of MN471010, ES&H Training Catalog. Compliance and additional training requirements shall be determined by the manager of 2552.

3.2 On-the-Job (OJT)/Qualification/Requalification

Operators working under this OP shall be properly trained before they are allowed to participate in the inerting activities described in this OP. The Department manager of 2552 shall determine if and when an employee in his/her department is adequately trained for these operations.

4.0

DEFINITIONS

- 4.1 Static Sensitive - the property of an explosive material or device which exhibits a discernible reaction when subjected to the test conditions described in SS302365 [Electrostatic Discharge Test Specification (Sandia Standard Man Model)].
- 4.2 Ground Plane Work Areas - areas and associated wrist straps that provide a means for maintaining static sensitive items in an electrostatic equilibrium.
- 4.3 Velostat bag or pillbox - a conductive container in which energetic material may be stored.
- 4.4 Inerting Cylinder- a heavy walled, stainless steel cylinder designed for the safe bakeout of small quantities of explosives and/or small explosive components. This cylinder (P/N: R45632) has been tested to verify that it will survive, undamaged, an internal detonation equivalent to 32 gm of TNT.
- 4.5 Primary Explosive - an explosive that can be made to detonate with a low level stimulus. These explosives are characterized by the fact that they are extremely sensitive to initiation by friction, temperature, shock, and/or electrostatic discharge. Examples: Lead Azide, Lead Styphnate and Barium Styphnate.
- 4.6 Secondary Explosive- An explosive that is less sensitive to initiation from the various energy sources than primary explosives. Examples: TNT, Tetryl, RDX, PBXs and Comp. C4.
- 4.7 Pyrotechnic Materials - physical mixtures of finely divided fuels and oxidizers, these may also contain various organic binders and color intensifiers. Once ignited, these mixtures can rapidly evolve a considerable amount of heat, light, and/or gas. However, not all

pyrotechnic materials evolve these by-products. This definition differs from the one given in MN471001 (ES&H Manual) which states that heat and gas are evolved and does not consider those pyrotechnics which do not evolve gas. Examples: $\text{TiH}_x/\text{KClO}_4$, B/CaCrO_4 , and Thermites.

- 4.8 Propellant - explosive composition used for generation of large quantities of hot gases. Primarily used for propelling projectiles and rockets.
- 4.9 Critical Temperature(T_c) - The DOE "Explosives Safety Manual" defines T_c as the: "Temperature above which the self-heating of an explosive causes a runaway reaction. It is dependent on mass, geometry and thermal boundary conditions."

5.0

HAZARDS

- 5.1 The explosives and explosive components to be inerted cover the range from igniters, with pyrotechnic materials, to extruded detonable explosive to pelletized high explosive powder.
- 5.2 Quantities of energetic materials inerted in the cylinder will be ≤ 20 grams TNT equivalent per trial. TNT equivalency for high explosives (HE) and HE components will be based on the HE's lead block test (Trauzel Test) equivalency to TNT, whenever possible.
- 5.3 There will be no significant explosion hazard to operating personnel during the inerting operations. The most significant hazard during this operation will be the burn hazard due to the high surface temperature of the steel cylinder.
- 5.4 Radiation hazards are due to the handling of metal parts which have been activated to levels on the order of nano-curies. The active nuclides include Ba-133, Co-56, 57, 58 and 60, Cs-134 and 137, Eu-154, K-40, Sc-46, Sn-113, Mn-54, Na-22, V-48, Y-88 and Zn-65. One sample is believed to be tritium contaminated to some very low levels, but no analysis has been done to confirm either the contamination or the levels.
- 5.5 There will be a compressed gas hazard due to the use of compressed N_2 for purging and inerting.

Warning

During the inerting operations the surface of the steel cylinder may reach dangerously high temperatures. Care must be taken to avoid any contact with the cylinder when it is at high temperature.

A SIGN SHALL BE INSTALLED IN FRONT OF THE CYLINDER DURING INERTING OPERATIONS. THE SIGN SHOULD PROVIDE A " Danger: Hot - Do Not Touch" WARNING.

IF THE CYLINDER IS TO BE HANDLED WHILE STILL HOT, THE OPERATOR(S) SHALL WEAR LEATHER GLOVES.

6.0 SAFETY EQUIPMENT

- 6.1 Conductive surfaces and tools shall be available for the purpose of packaging/unpackaging explosives and explosive components. This area(s) shall be used for operations during which the explosives are not contained in appropriate, closed containers.
- 6.2 Approved safety glasses shall be worn at all times by both operators and/or casuals while operations are underway in the laboratory.
- 6.3 An approved wrist strap shall be used during all operations involving static sensitive explosives and explosive components and shall be visually inspected before use to ensure that it is properly connected and is not damaged.

7.0 STANDARD OPERATING PROCEDURE**General Explosives Operating Guidelines**

This OP applies to the personnel in Department 2552 or other personnel who handle explosives, explosive components, propellants, and pyrotechnics in the aforementioned laboratory. This OP is intended to address explosive handling ES&H requirements for this site during operations to thermally inert explosives.

7.1 Guidelines

- 7.1.1 The smallest amount of explosive necessary to complete the task shall be used.
- 7.1.2 The departmental "Two Man" rule (Appendix A) shall be used for all operations involving energetic materials.
- 7.1.3 Persons handling static-sensitive explosives and explosive components should keep the electrical potential at the same level between themselves, the components, and the next assembly. *This can be accomplished by wearing a wristband grounding strap securely attached to a ground that is common to the explosive component and the next assembly or the use of a conductive ground plane with a wrist grounding strap.*
- 7.1.4 An approved wrist strap shall be used during all operations involving static sensitive explosives and explosive components and shall be visually inspected before use to ensure that it is properly connected and is not damaged.
- 7.1.5 Static sensitive items shall not be handed from one person to another. *The component shall be placed on a conductive surface so that the second person can pick it up.* This insures that both the operator and the item are at the same electrical potential.
- 7.1.6 For the inerting operations, the maximum quantity allowed in process at any one time is limited to 20 grams TNT equivalent of explosives for each inerting run.
- 7.1.7 The maximum quantity allowed in the laboratory at any one time is limited to 100 grams TNT equivalent of explosives. Any requirement to exceed this limit shall require the approval of Safety Engineering, Department 7732 and the managers of 2552 and 14621. *Only storage cabinets that comply with DOD Explosive Safety Standards and are approved by Safety Engineering will be used to store explosives.* In no case shall the quantity of explosives exceed the maximum quantity approved for the storage cabinet.
- 7.1.8 Conductive surfaces and tools should be kept free of dust.
- 7.1.9 When working with energetic materials or explosive components all personnel, including casual observers, shall wear safety glasses.

- 7.1.10 MSDSs will be maintained for all energetic materials and explosive components if available (some older components and energetic materials do not have MSDSs). If no MSDS is available, the hazards associated with the materials will be described, to the extent possible, in writing and based on the operators expertise and knowledge of the materials. A copy of this description will substitute for an MSDS.
- 7.1.11 All laboratory operations are performed in a protected building and do not have to stop during electrical storms or high potential gradient conditions.
- 7.1.12 All instruments will be used in accordance with the manufacture's recommendations (user's manuals or application notes) unless an OP is written and approved.

7.2 Emergency Procedures

- 7.2.1 Working alone with explosives is strictly prohibited. Observe the "Two Man Rule".
- 7.2.2 In the event of an emergency involving one individual, the second person shall render immediate assistance as required, e.g., perform emergency shut down procedures if possible and summon outside assistance if needed.
- 7.2.3 In the event of a laboratory or area emergency, the proper procedure will be to activate the Area V alarm, kill the power to the inerting operation and follow all Area V emergency procedures. In the event that an Area V alarm is sounded from elsewhere in Area V, the proper procedure will be to kill the power to the inerting operations and then follow all Area V emergency procedures.
- 7.2.4 Contact Department Managers, 2552 and 14621 immediately following any emergency involving the inerting operations.
- 7.2.5 Contact Department 7714 for additional safety or ES&H information.

7.3 Inerting Procedure

- 7.3.1 Materials Needed
- 7.3.2 Inerting cylinder (P/N: R45632) and associated hardware
- 7.3.3 Temperature monitor and controller
- 7.3.4 Data logger
- 7.3.5 Heating pan and tubes of various sizes, Al foil.

- 7.3.6 Ground plane work area and wrist straps
- 7.3.7 Nitrogen gas cylinder with regulator
- 7.3.8 Vacuum pump
- 7.3.9 In-line HEPA filter
- 7.3.10 Moisture traps and charcoal filters.
- 7.3.11 Conductive plastic gloves.
- 7.3.12 Chemical Fume Hood

7.4 Inerting of Explosives and/or Explosive Components.

Note: The following operations should be performed while wearing PVC or rubber gloves. All operations during which explosives are being handled also require that the person handling the explosive be grounded via a wrist strap.

- 7.4.1 Verify that the electrical power cable from the temperature controller to the inerting cylinder is disconnected. Remove the uninstrumented cover plate from the steel cylinder and extract the heating pan for easy access. If the insulating blankets are in place around the pan, remove them and place them on a pre-prepared, paper covered area.
- 7.4.2 Prepare the explosive or explosive component for treatment as described in Appendix B. This assembly may be pre-prepared at an appropriate site prior to beginning Section 7.4 of this OP.
- 7.4.3 Place the explosive assembly from step 7.4.1 into the heating pan.
- 7.4.4 Install the heating pan into the cylinder; being careful to:
 - 7.4.4.1 insert the controlling thermocouple inside the tube or foil package or above the sample in the foil boat. Replace the heating pan cover.
 - 7.4.4.2 install the insulating blanket over the heating pan.
- 7.4.5 Attach the closure plate onto the cylinder with the bolts provided. Torque the bolts into the cylinder to 30 in-lbs.
- 7.4.6 Purge the cylinder prior to application of heat.
 - 7.4.6.1 Verify that the valve from the N₂ cylinder to the inerting cylinder is closed.

- 7.4.6.2 Open the valve from the vacuum pump to the inerting cylinder and start the vacuum pump.
- 7.4.6.3 Pump out the cylinder to ≥ 20 inches of Hg vacuum, then close the valve between the cylinder and the pump.
- 7.4.6.4 Back fill the cylinder with N_2 gas to ambient pressure.
- 7.4.6.5 Repeat steps 7.4.6.1 - 7.4.6.3

7.4.7 Attach electrical power cable from the temperature controller to the inerting cylinder.

7.4.8 Install temperature hazard warning sign on cylinder.

7.4.9 The T_c data provided in Appendix C is for information purposes; the temperature(s) used here will be well in excess of those data. Set the temperature controller to a 500°C set point. Start the data logger to collect temperature data and begin the heating of the explosives. Observe the temperature rise of the heating block to assure that the set point is reached. Allow the heating to continue for $\geq$ two (2) hours.

Note: The Inerting Cylinder (P/N 45632) has been designed, and tested to a 125% overtest level, to safely contain 25 gm TNT equivalent; the inerting system as currently configured does not have an upper limit shutoff. The DOE Explosives Safety Manual, Chapter II, Paragraph 21.3 "Heating Operations", exempts "Systems capable of total containment of the effects of an explosion..." from the requirement for continuous monitoring and/or override shutoff protection. In the event of a runaway temperature excursion there are two additional fail safe features: 1) the heating elements are rated for temperature to 600°C only, the elements should fail in a runaway event; and 2) the high explosives all decompose and/or detonate, i.e. exotherm, at temperatures well below the 500°C set point level. In the event of a runaway the explosives will be destroyed before the cylinder reaches a temperature where its physical properties are compromised. The only hazard in such an event will be a high temperature hazard.

7.4.10 Cease application of heat to the heating pan. Stop data logger and download the data to the notebook PC for later analysis.

Note: At this point the explosive hazard has been removed from the inerting cylinder, unattended, i.e. overnight, cooling is permitted.

7.4.11 Allow the inerting cylinder to cool to ambient room temperature.

7.4.12 Purge the cylinder of reaction product gases.

- 7.4.12.1 Verify that the valve from the N₂ cylinder to the inerting cylinder is closed.
- 7.4.12.2 Open the valve from the vacuum pump to the inerting cylinder and start the vacuum pump.
- 7.4.12.3 Pump out the cylinder to ≥ 20 inches of Hg vacuum, then close the valve between the cylinder and the pump.
- 7.4.12.4 Back fill the cylinder with N₂ gas to ambient pressure.
- 7.4.12.5 Repeat steps 7.4.12.1 - 7.4.12.4

7.4.13 Disconnect the power cable from the controller to the cylinder; disconnect the power cable at the controller. Remove the uninstrumented cover plate from the cylinder.

7.4.14 Remove the heating pan from the cylinder and remove the tube(s) and/or the foil container from the pan. Place the tube and/or foil container, with the inerted, formerly explosive hardware, in a hazardous, mixed waste container.

7.5 Post Test Analysis

Prepare the in-line filter, the in-line HEPA filter and a sample of the vacuum pump oil for radioactive contamination analysis. Prepare a swipe of the inside of the inerting cylinder for radioactive contamination analysis.

8.0 WASTE DISPOSAL

There will be no generation of explosive waste in this operation. All wastes generated will have been decontaminated for explosives. The wastes generated from these operations will be disposed of as hazardous or mixed waste.

9.0 ES&H REPORTING AND DOCUMENTATION

All previously explosive, mixed waste materials and components which will be residual to the inerting operations shall be treated as hazardous, mixed waste. All the applicable documentation and records for management of such waste shall be maintained.

- 9.1 Inspection records of all ES&H related equipment shall be maintained as required in the SNL ES&H Manual (MN471001)
- 9.2 Explosive inventory records shall be maintained in accordance with Department 2552 requirements.
- 9.3 Personnel training records shall be maintained in the office of Department 2552.
- 9.4 The generic ES&H Standard Operating Procedure for Reporting Procedures for Accidents, Incidents, Occurrences and Releases for Sandia National Laboratories (SOP470041) shall be followed.

10.0 REFERENCES

- 10.1 Sandia Explosives Safety Manual, MN471011, Issue A, 12/3/93.
- 10.2 DOE Explosives Safety Manual, DOE/EV/06194, Revision 7
- 10.3 Environment, Safety & Safety Manual MN471001
- 10.4 ES&H SOP SP472526
- 10.5 ES&H SOP GN470041

11.0 APPENDICES

- Appendix A: Two Man Rule
- Appendix B: Assembly of Explosives and Explosive Components for Inerting in Cylinder P/N R45632
- Appendix C: Critical Temperatures of Some Explosives.

12.0 AUTHORIZED USERS LIST

The following personnel are authorized to perform activities in Building 6588, the HCF, in accordance with this procedure. Their signature hereby indicates they have read, understood, will conform to all requirements of this procedure, and operate within the stated constraints.

[illegible]

- frequently monitor the Operator
- provide assistance as directed by the Qualified Operator
- know the emergency/accident procedure(s)
- provide prompt and easily understood communications
- be familiar with the facility hazards
- be familiar with the use of safety equipment in the facility
- perform emergency shut down procedure(s) as required

for activities in Building 6588, the HCF, in accordance with this procedure. By their signature they hereby indicate that they have read, understood and will conform to all requirements of this procedure. Signing this sheet does not qualify the personnel as Operators, but rather as "buddies" for **ONLY** the above mentioned location.

ORG./DATE

[illegible]

Appendix A

"Two Man Rule" Departments 2552, 2553 and 2554

The following definition for a departmental "Two Man" rule is based on the requirements of the DOE explosives safety manual and Air Force regulations. DOE requires that for all explosive handling operations;

"personnel shall be assigned in a manner such that each worker's presence is frequently monitored and assistance can be provided or aid summoned in the event of an emergency."

Further, the DOE requires that;

"no person shall work alone performing explosives activities that have a high risk of serious injury. Prompt and easy communications with other employees shall be provided."

The Air Force requires that while performing any explosive operation;

"at least two persons are present so that one may give assistance to the other if an emergency occurs "

These requirements point to the need for any explosive operation to have a monitor who is not directly participating in the operation. The following definition for the department "two man" rule is based on these requirements.

Any explosive operation shall be monitored by at least one person who is not a participant in the operation. This monitor shall be at a distance such that in the event of an emergency the monitor is not endangered by the emergency and will be able to render assistance as needed and call for additional help as required. Monitoring may be performed by direct visual or electronic (audio or video) means. If an electronic monitoring system is used it must not be capable of being interrupted by a third party. The monitoring need not be constant but may be at intervals suitable for the hazard level of the operation being performed. Before commencing any explosive operation a monitor must be identified by the person performing the operation.

In the event of an emergency the monitor shall render immediate assistance as required (e.g., perform emergency s u own procedure, if possible, etc.) and summon outside assistance if needed (e.g. emergency medical assistance, fire fighting personnel, etc.).

Appendix B
Assembly of Explosives and Explosive Components
for Inerting in Cylinder P/N R45632

A.: Assembly of Components:

Detonators and/or igniters will be loaded into steel tubes for inerting. The components will be held in place in the tube by plugs of steel wool. As much as possible, the component will be "aimed" at the tube wall. Figure A, in which an MC4217 detonator is shown loaded in a tube for illustrative purposes, shows the intended packaging. After treatment, the tubes can be unloaded by extracting the steel wool plugs and the components using tweezers.

B.: Assembly of Bulk Explosives:

Bulk explosives, either loose powder or free standing pressed pellets, will be placed and wrapped in Al foil. The foil will be folded such as to form a semi-sealed package; this will help keep most of the carbonized material contained within the package for disposal. These foil packages can be placed into tubes if size permits, but it is not necessary to do so.

C.: Assembly of Explosively Configured Polycarbonate (MC2370):

The explosives loaded polycarbonate assemblies will be placed into an Al foil "boat" which will be of sufficient size to contain ~ twice the volume of the polycarbonate block(s). The polycarbonate blocks have a pellet side and a track side, the track side is the side on which the explosive is extruded; the pellet side is the side which shows the ends of the pressed pellets. The block(s) will be placed into the boat with the pellet side down. The polycarbonate blocks can be treated in groups of two. After treatment the foil will be folded over the re-solidified polycarbonate residues to form a package for disposal.

Appendix C Critical Temperatures of Some Explosives

The conditions to which the explosive materials will be subjected during inerting operations described in this OP will be very similar to conditions existent in a Differential Scanning Calorimeter (DSC). Consequently, under the conditions of those operations and consistent with the definition of T_c , the critical temperature (T_c) of the explosives will be considered to be the same as the "Onset of Exotherm" temperature measured by DSC.

Unless otherwise indicated, the %TNT values for these explosives have been taken from Explosives, 3rd Ed., Rudolf Meyer, VCH, 1987.

<u>Explosive</u>	<u>Atomic Composition</u>	<u>DSC Mol. Wt. (gm/mole)</u>	<u>Minimum Melt. Pt. (Deg. C)</u>	<u>Exotherm^a Temp. (C)</u>	<u>Bakeout Temp. (C)</u>	<u>%TNT Trauzel</u>
PETN	$C_5H_8N_4O_{12}$	316.1	141	150	210	174
β -HMX	$C_4H_8N_8O_8$	296.2	280	235	295	160
RDX	$C_3H_6N_6O_6$	222.1	204	200	260	160
HNAB	$C_{12}H_4N_8O_{12}$	452.2	221	265	325	
HNS	$C_{14}H_6N_6O_{12}$	450.1	318 ^b	295	355	100
TATB	$C_6H_6N_6O_6$	258.1	350	335	395	60
TETRYL	$C_7H_5N_5O_8$	287.2	129.5	187	247	125 ^e
TNT	$C_7H_5N_3O_6$	227.1	81	260	320	100
CP	$C_2H_{15}N_{10}O_8Cl_2Co$	437.0	Dec. ^c	240	300	
TiKP	Ti/KClO ₄	Blend	Dec. ^d	430	490	N/A
TiH _x KP	TiH _{1.65} /KClO ₄	Blend	Dec. ^d	370	450	N/A

Notes:

*: This data is from DTA, LANL Explosives Handbook.

a: These data represent the temperature at which these materials begin a self-sustaining, irreversible decomposition, either deflagration or detonation.

b: HNS undergoes decomposition as it melts: Rudolf Meyer, Explosives, 3rd Ed., VCH, 1987.

c: CP does not melt, it decomposes violently at the given temperature.

d: TiKP and TiH_xKP, in its various H_x forms, do not melt, they auto-ignite and deflagrate rapidly when heated to the given temperature.

e: From AMC Pamphlet: AMCP706-177; January, 1971.

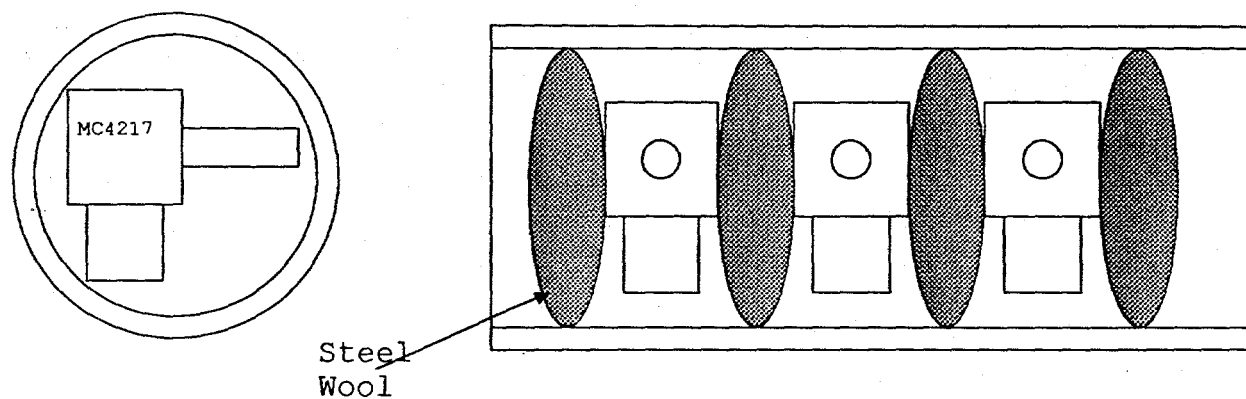


Figure A. : Component Loaded into Inerting Tube

Distribution:

	MS 0953	W. E. Alzheimer, 1500
	MS 1452	J. G. Harlan, 1552
5	MS 1452	V. M. Loyola, 1552
5	MS 1452	S. D. Reber, 1552
	MS 1315	T. E. Blejwas, 7500
	MS 1303	J. R. Guth, 7573
	MS 1303	G. L. Nordyke, 7573
5	MS 1303	P. K. Peterson, 7573
	MS 1142	D. T. Berry, 6521
	MS 1142	D. J. Bragg, Jr., 6521
	MS 0720	S. W. Longley, 6626
	MS 9018	Central Technical Files, 8523-2
5	MS 0899	Technical Library, 4414
	MS 0619	Print Media, 12615
2	MS 0100	Document Processing, 7613-2 for DOE/OSTI